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THE RELATIONSHIP BETWEEN TREE-CORE AND GROUNDWATER

TRICHLOROETHYLENE CONCENTRATIONS FOR

GROUNDWATER PLUME DELINEATION

by

Kerry L. Lewis

A thesis submitted in partial fulfillment
of the requirements for the degree

of

MASTER OF SCIENCE

in

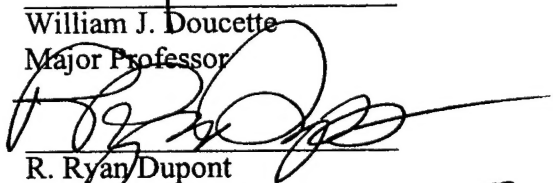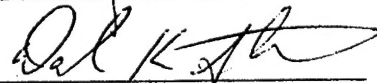Civil and Environmental Engineering

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2001

ABSTRACT

The Relationship Between Tree-Core and Groundwater Trichloroethylene

Concentrations for Groundwater Delineation

by

Kerry L. Lewis, Master of Science

Utah State University, 2001

Major Professor: Dr. William J. Doucette
Department: Civil and Environmental Engineering

A 17-month field study designed to evaluate the feasibility of using trees as a cost-effective groundwater sampling and plume delineation tool was recently completed at site Operable Unit 4 at Hill Air Force Base (Ogden, UT). Using a hand-driven incremental borer, tree-core samples were collected monthly from cottonwood (Populus deltoides), russian olive (Elaeagnaceae elaeagnus), poplar (Salicaceae populus), apple (Malus pumila), and box elder (Acer negundo) trees located within and outside a trichloroethylene (TCE)-contaminated shallow groundwater plume. Concentrations of TCE in the core samples were determined using a headspace gas chromatography procedure. Variations to the headspace analysis method were made and results compared to other methods of TCE measurement for performance comparison. A relationship between tree core (0.001 to 32 mg/kg) and groundwater (0.2 to 4890 mg/L) TCE concentrations was observed (e.g., trees located above areas of high groundwater TCE

concentration were found to contain high TCE concentrations). Stable isotopes of hydrogen were used to show that shallow TCE-contaminated groundwater is the most likely source of water used by the trees. Wood sorption isotherms (using site specific trees) were completed using $[^{14}C]$ TCE in a sealed batch process. Wood-water partitioning coefficient (K_{wood}) values of 16.42 mL/g (cottonwood) and 11.51 mL/g (russian olive) were obtained from linear isotherms. When measuring TCE concentrations in water, these K_{wood} values can now be used to estimate original concentrations of TCE within the tree tissues. Desorption coefficients were measured through sequential dilution steps and found to be reversible along the sorption isotherm. Reversible desorption suggests that desorption occurs in a short period of time (sorption experiments were completed in a 24-h time frame); thus, measured concentrations within a tree tissue should represent groundwater taken by a tree within the same 24-h time period.

(201 pages)

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Upon arrival and after having received an incredible sponsorship under the U.S. Air Force Institute of Technology Civilian Institute program for this master's degree program, I was searching hard for a project with tangible benefits for the Air Force. I want to extend my sincere appreciation to Bill Doucette for permitting me to join his Hill Air Force Base phytoremediation research team and for his incredible patience with the countless interruptions I made seeking his advice. I would not have been able to accelerate and finish this incredible journey without his selfless diligence, support, and understanding.

I would also like to thank the remaining members of my committee, Bruce Bugbee, R. Ryan Dupont, and David Stevens, for their advice, patience, and understanding of my looming deadlines. Having earned my undergraduate degree at Texas A&M University, my "big head" was quickly humbled by the incredible faculty that is the Environmental Engineering Division at Utah State University, of which my committee members were shining examples. Not only was your support during the creation of this thesis appreciated, I want to thank you for your challenging lectures and your ability to share information in a way that even I could understand.

To those others in the trenches with me, Chris Pajak, Scott Benson, and Nathan Zaugg, the heartiest of thanks for your friendship, technical advice, and for your sense of humor. Best of luck to you as you finish your degree programs and take on the new challenges that await beyond Logan, UT.

I am extremely grateful to Jon Ginn who has not only been my best friend and mentor here in Logan, but also my main support point of contact at the Environmental

Management Directorate, Hill Air Force Base. Thank you for getting the additional OU4 well sampling on contract for Spring 2001—this thesis would have been mute without that groundwater data.

How could I have ever accomplished this program without my precious, beautiful wife, ~~████~~? She promised her full support prior to my acceptance of this degree program and then sacrificed immeasurably during its fruition. The peace you brought to our home, the sacrifices you made, and the faithful prayers you offered on my behalf I will never forget.

Kerry Lewis

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CHAPTER 1

INTRODUCTION

Background

Jordahl et al. (1997) defined phytoremediation as the use of plants to remove environmental pollutants from contaminated sites. It is an emerging technology that is being promoted as an effective and inexpensive cleanup method for shallow groundwater and soil contaminated with chlorinated solvents like trichloroethylene, commonly known as TCE (Nietch et al., 1999). Plant uptake, resulting in metabolism and/or phyto-volatilization, has been identified as one of the potentially important phytoremediation mechanisms for TCE. However, even as phytoremediation is being applied at many TCE-contaminated sites, quantifying the magnitude of uptake has proven to be difficult, especially in a field setting.

The uptake of organic contaminants like TCE by plants is thought to be a passive process by which the contaminants move into the plant along with the water taken in by the roots. Laboratory studies have generally shown that uptake is a function of the amount of water transpired by the plant, the hydrophobicity of the chemical expressed as the octanol/water partition coefficient (K_{ow}), and the concentration of chemical in the water used by the plant (Briggs et al., 1982; Bromilow and Chamberlain, 1995; Burken and Schnoor, 1998; Cunningham et al., 1996; Orchard et al., 2000). This suggests that for a given chemical, the amount of plant uptake in a field setting, and most likely the concentration in the plant, should be a function of the concentration of the chemical in the groundwater used by the plant. One implication of this possible relationship is that plants

could be used as natural groundwater pumps instead of more expensive wells. In this thesis project, the possibility that trees might be used as an efficient and inexpensive water sampling tool for efficient, inexpensive site characterization of the range and magnitude of contaminant plumes was investigated.

Assuming trees are growing at the appropriate locations, the collection of tree cores may simplify groundwater monitoring programs. Tree cores can be collected in less than 1 minute using an incremental borer. Contrast this easy sample collection method with the logistics, time, and equipment costs required to install groundwater sampling wells. Associated cost savings (attributed to a reduced need for groundwater wells) can be realized if a relationship between groundwater contamination and tree core concentration can be better defined.

For proper site characterization using tree cores, a quantitative relationship was sought between tree core and groundwater contaminant concentrations using mature, indigenous trees growing above a TCE-contaminated shallow groundwater aquifer at Operable Unit 4 (OU4) at Hill Air Force Base (HAFB), UT. This site provided an opportunity to investigate mature indigenous trees in a natural field environment.

Headspace analyses followed by gas chromatography using an electron capture detector (GC/ECD) have shown great potential for quick and quantitative evaluations of TCE and similar compounds (e.g. volatile, chlorinated) in tree tissues (Doucette et al., 2000). Preliminary method development was accomplished by Pajak et al. (2000) during phytoremediation research as Operable Unit 2, another site located at HAFB. To evaluate tree tissue concentrations of TCE, further advancement, testing, and validation of this quick and quantitative collection and analyses method was deemed necessary.

Although headspace/gas chromatography may be a suitable method for the rapid analysis of tree core and leaf samples (Doucette et al., 1998; Pajak et al., 2000; Vroblesky et al., 1999), the procedure's effectiveness and reproducibility have not been adequately evaluated. The only other literature found to date (Vroblesky et al., 1999) regarding headspace/gas chromatography for the evaluation of tree cores describe results useful only for the qualitative evaluation of the presence and general magnitude of TCE within the plant tissue. Quantitative techniques and evaluations are therefore necessary to determine the significance of phytoremediation's contaminant removal mechanisms.

Cunningham et al. (1996) stated that "...phytoremediation R & D represents an excellent area in which basic and applied science have begun to work together to reach a potentially valuable technology." This thesis research carries the spirit of Cunningham's statement. Data collected and methods developed during this investigation are beneficial to the research of those interested in phytoremediation and in the measurement of tree tissue concentrations of groundwater contaminants.

Objectives

The overall objective of this thesis was to investigate the relationship between TCE tree core concentrations and TCE groundwater concentrations as a potential method for groundwater plume delineation. Hill AFB OU-4 was an excellent site for this research because it contains mature trees above a shallow TCE-contaminated aquifer and its semiarid environment promotes groundwater use. Specific secondary objectives included:

1. Development of a headspace analysis method for the quantitative determination of

TCE concentrations in plant tissue samples.

2. Comparison of the developed headspace method with purge and trap.

3. Identification of seasonal tree core concentration variations.

4. Initial investigation of the potential use of stable isotope measurements at HAFB to determine the fraction of groundwater used by the trees.

Site Characterization

Hill AFB is located approximately 25 miles north of Salt Lake City, UT. The base covers approximately 6,700 acres and is located on the Weber Delta, a terrace approximately 300 ft above the surrounding valley floors of Weber and Davis Counties (Fig. 1-1). OU4 is located near the northern boundary of the base and consists of the north gate dump area, the spoils pit, the munitions dump, and landfills 1 and 2. The terrain consists of alternating gentle and steep slopes dropping from the upper terrace, crossing an irrigation canal, then dropping down to the valley floor approximately 300 ft below the upper terrace. The steep slopes promote groundwater seepage during wet periods and generally promote shallow depths to the groundwater table. Easy access to groundwater along steeper slopes has promoted the substantial growth of various tree species that include apple, willow, russian olive, poplar, box elder, and cottonwood. Most of these areas containing trees may be classified as riparian corridors due to their location near seeps or periodic springs (Ehleringer et al., 1991). The area climate is described as semiarid with an average rainfall of less than 20 inches/year. Snowfall can be significant in the winter and provides recharge for the shallow aquifer during the winter months.

Contaminants detected at OU4 consist of volatile organic compounds (VOCs)

including benzene, chloroform, 1,1-dichloroethene (DCE); cis- and trans- 1,2-DCE;

1,2-dichloroethane (DCA); methyl ethyl ketone (MEK); tetrachloroethene (PCE);

toluene; trichloroethylene (TCE); and xylenes; as well as metals including arsenic,

barium, boron, cobalt, nickel, and selenium. Contamination is the result of decades of
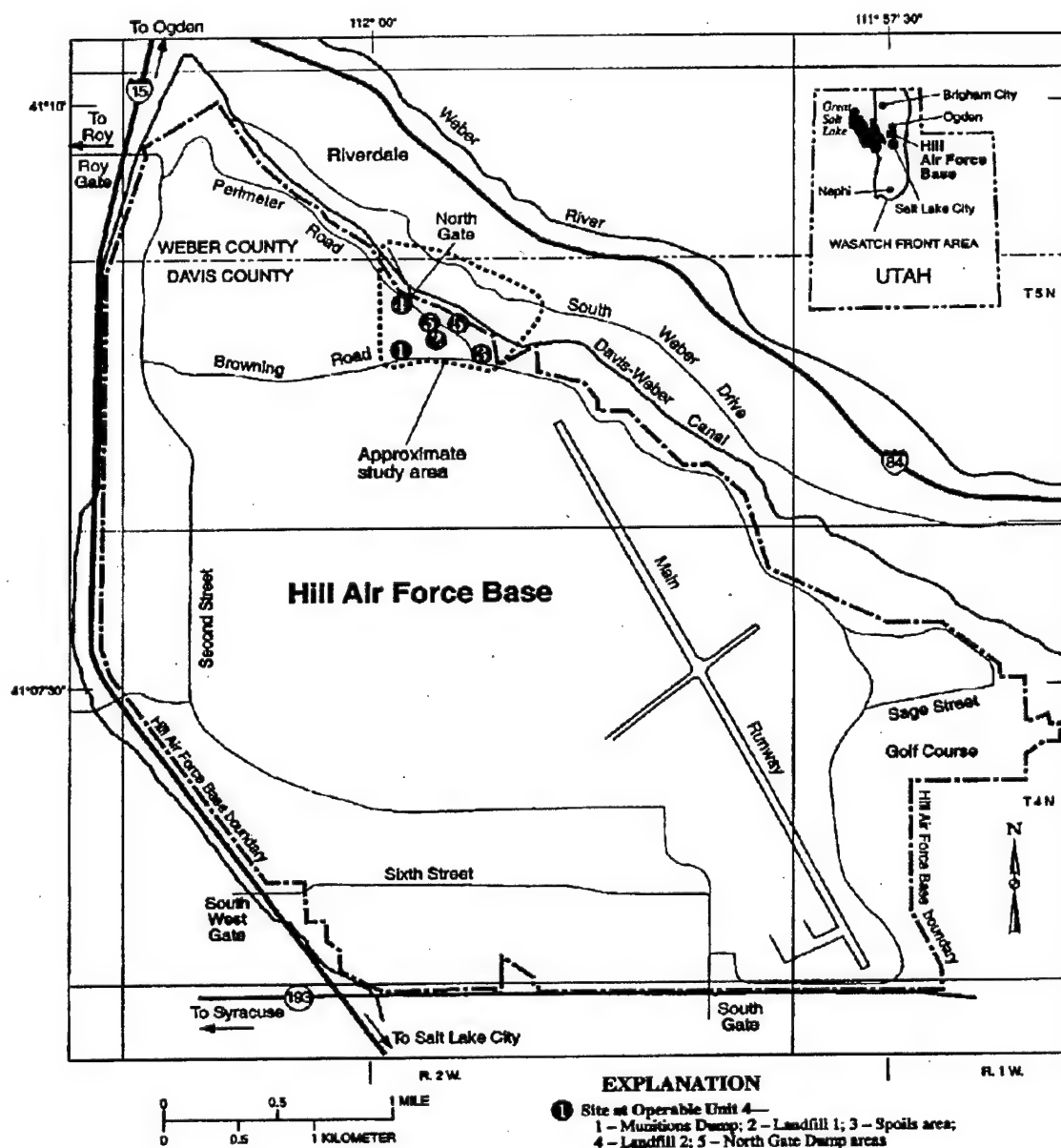
Fig. 1-1. Location of Hill Air Force Base site OU-4 (Haraden, 1999, pg. ES-1).

improper disposal of waste liquid solvents and degreasers. VOCs have been detected in groundwater, seeps, and soils; metals have been detected only in groundwater. TCE is the principal contaminant at OU4 being the only VOC consistently detected in groundwater and seeps at concentrations that exceed the maximum contaminant level (MCL) of 5 μg/l. The area contaminated by TCE is approximately 69 acres as determined from analytical results of samples collected through July 1999. Fig. 1-2 shows the general location of the contamination source (landfill 1), the extent of TCE movement, and the sites selected for this research. Most TCE contamination has been detected in the upper 30 ft of the shallow aquifer (ideal for potential use by deep-rooted trees) with depths to groundwater varying from 5 to 35 ft. Seeps and springs occur where layers that are more permeable intersect the hillside conducting water to the surface. Soil
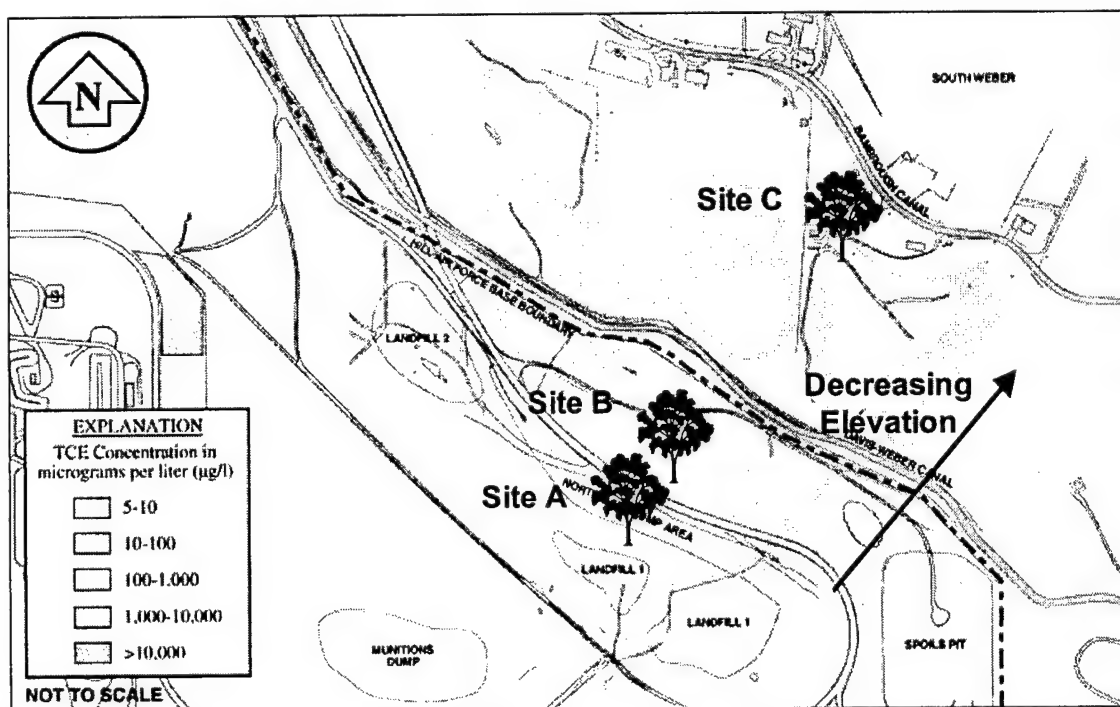
Fig. 1-2. Hill Air Force Base site OU-4. Tree symbols identify the sampling sites located above high, medium, and low groundwater TCE concentrations.

consists of heterogeneous, finely interbedded silt, clay, and sand, with occasional thicker

sand, silt, and clay layers. General direction of regional groundwater flow is to the north-

northeast following the topography of the hillside. Lateral heterogeneity and probable

preferential flow paths in the sediments beneath OU4 are expected to create large

deviations from the generally expected regional flow direction.

Horizontal conductivities in the sediment are several orders of magnitude higher

than the vertical components with the predominant direction of groundwater flow being

horizontal (Montgomery Watson and Utah State University, 1999). Site characterization

information was taken from Montgomery Watson and Utah State University (1999) and

US Air Force (1999)

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CHAPTER 2

LITERATURE REVIEW

TCE Characteristics

TCE (CAS# 79-01-6) is a nonflammable, colorless, recalcitrant, volatile compound often used as a solvent for cleaning metal parts. TCE has also been used in adhesives, paint removers, typewriter correction fluids, and spot removers. Past TCE use was extensive at Hill AFB. An excellent parts cleaner and degreaser, TCE waste often contained trace amounts of other oil and grease products resulting from its use as a solvent. Poor disposal practices (landfills at the base boundary) resulted in its eventual contamination of the shallow groundwater at OU4. TCE is not thought to occur naturally in the environment [Agency for Toxic Substances and Disease Registry (ATSDR), 1997].

Physical constants of TCE most pertinent to this research are listed below:

1. Molecular Weight (g): 131.388
2. Melting Point (°C): -84.7 (1 atm)
3. Boiling Point (°C): 87.21 (1 atm)
4. Density (g/mL): 1.4642 (20°C)
5. Vapor Pressure: 60 mm Hg at 20°C [Agency for Toxic Substances and Disease Registry (ATSDR), 1993]
6. Log K_{ow}: 2.53 (Mackay et al., 1992)
7. Aqueous Solubility (-log C_w^{sat}, mol/L): 2.04 (Schwarzenback et al., 1993)
8. Log K_h (L-atm/mol, 25°C): 1.03 (Mackay et al., 1992)

Reference unless otherwise noted: (CRC Press, 2000)

Acute and chronic (long-term) inhalation exposure to TCE in humans results in central nervous system (CNS) effects, with symptoms such as dizziness, headaches,

confusion, euphoria, facial numbness, and weakness. Ironically, TCE was once used as a general anesthetic [Agency for Toxic Substances and Disease Registry (ATSDR), 1993]. The US EPA considers TCE as an intermediate between a probable and possible human carcinogen (Group B/C) and is in the process of revising its cancer risk assessment guidelines (USEPA, 2001).

Plant Uptake of TCE/Phytoremediation

Any compound reaching the soil may be subject to sorption into soil organic matter, decreasing that portion available for plant uptake. Doucette et al. (1999) reported large differences (10X-100X) between sorption and desorption coefficients for soil packages taken from site OU4. If other environmental processes like volatilization or biodegradation are faster than soil desorption, then soil desorption could limit the availability of a compound for plant uptake. Regardless of the degree of availability of a compound for plant uptake, the ultimate factor in plant uptake is the passage of a compound through plant membranes.

Plant structure influences the uptake and transport of xenobiotics like TCE from pore water in the rhizosphere into the root structure. Compounds entering plant roots can move in the intercellular spaces of the cortex (a part of the apoplast) until they reach the endodermis (see Fig. 2-1). The endodermis is a cylindrical sheath of cells that are tightly bound together by the lignified Casparian strip wall. This wall surrounds the vascular tissues in the stele. In order to pass the endodermis, a chemical must enter the endodermal cells first. Once in the endodermal cells, the chemical has access to the vascular tissues of the phloem and xylem (Bromilow and Chamberlain, 1995).

Differences in pH exist among the various compartments of plants (e.g.,

xylem~5.0, phloem~8.0, cell vacuoles~5.5, and cytoplasm~7.5). Although these

differences do not influence the distribution of non-ionized compounds (like TCE), they

can have strong influences on ionized compounds resulting in ion trapping. Ion trapping

can be defined as the separation of dissociated and undissociated forms of a compound by

the action of a selective membrane that maintains differing pH across its boundary

(Bromilow and Chamberlain, 1995).

According to Bromilow and Chamberlain (1995), the most important property

controlling the movement of nonnutrient organic chemicals in plants is lipophilicity.

Lipophilicity, a measure of a chemical's affinity for the aqueous phase versus an affinity

for lipid-like phases, determines movement across plant membranes. Once across plant

membranes, the chemical is available for transport via the phloem and xylem. However,

partitioning and/or sequestration of compounds onto plant solids can limit this movement
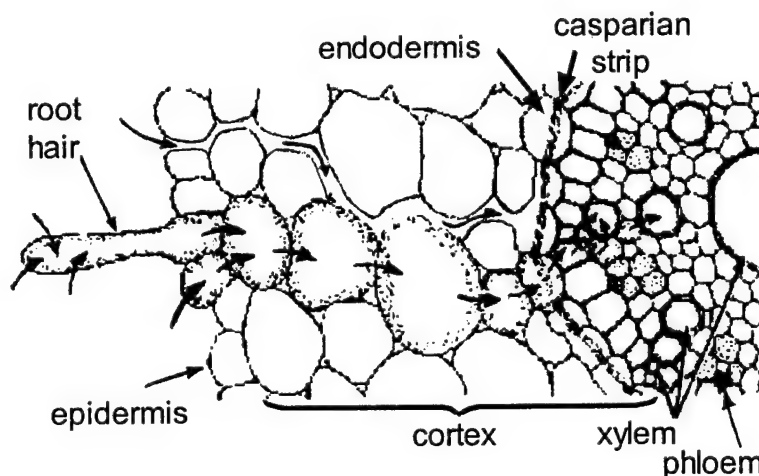
Fig. 2-1: Wheat root cross section showing possible routes of water and solute movement into the plant. (Adapted from Esau, K. 1958. *Plant Anatomy*. John Wiley & Sons, New York, p. 506)

via the phloem and xylem. This limitation of movement across membranes is also a function of lipophilicity. The transpiration stream concentration factor (TSCF) is a measure of the efficiency of this movement of a contaminant from the roots to the shoots via the transpiration stream – a ratio of the concentration in the transpiration stream to the concentration of the chemical in the external soil solution.

The term TSCF was first defined in 1974 for use in the comparison of chemical partitioning within plant species (Shone and Wood, 1974). As TSCFs approach unity, the compound is said to move with the same efficiency of water (unless significant degradation is occurring within the plant). Significant work on the relationship between lipophilicity (estimated using log K_{ow}) and TSCFs has shown that compounds having log K_{ow} values around 1.8 [for barley shoots; (Briggs et al., 1982)] and 3.07 [for de-topped soybean plants; (Hsu et al., 1991)] had optimum TSCF values.

Bromilow and Chamberlain (1995) reported that as log K_{ow} is decreased the TSCF decreases because polar compounds will have difficulty passing through the hydrophobic membranes. Furthermore, they also reported that the more lipophilic compounds cross the endodermis with much less efficiency than water, resulting in a TSCF decrease as log K_{ow} is increased beyond the optimum log K_{ow} value. Although Bromilow and Chamberlain (1995) did not offer an explanation for the optimum log K_{ow} differences, Orchard (1998) suggested that the differences between these two optimum log K_{ow} values may be a result of differences in plant age, plant size, or experimental technique. Briggs et al. (1983) conducted controlled growth greenhouse experiments using barley shoots and radiolabeled compounds. They concluded that constant maximum concentrations in stems were rapidly attained (24-48 h) for chemicals of log

K_{ow}<3, but were attained more slowly for more lipophilic chemical. The log K_{ow} for

TCE has been reported to be approximately 2.53 (Mackay et al., 1992). Consequently,

detectable levels of TCE (log K_{ow} 2.53) in the tree cores were expected.

Although literature describing the magnitude of uptake and phytovolatilization of

TCE is inconsistent, there is widespread belief that plant uptake and phytovolatilization

are significant. This belief has provided the motivation for additional sampling. Results

obtained from several separate studies at HAFB's OU2 and OU4 clean-up sites by Utah

State University have determined that either significant uptake must be presently

occurring or past tree-core TCE contamination is present and not degrading to achieve

the high TCE tree-core concentrations recently measured.

Two recent studies conducted at Cape Canaveral Air Station (CCAS) in Florida

and HAFB in Utah (Doucette et al., 1998, 2000; Hayhurst, 1998) specifically studied the

fate of TCE and metabolic by-products in trees growing above shallow contaminated

groundwater. Both locations contained similar groundwater concentrations of TCE and

similar depths to groundwater. Precipitation patterns and tree species highlight their

differences. CCAS lies within a subtropical climate region (>50 inches precipitation per

year) while Hill AFB lies within a semiarid climate region (<20 inches precipitation per

year). The CCAS study involved live oak, castor bean, and saw palmetto plant species

while the Hill AFB study involved poplar, willow, and russian olive. Doucette et al.

(2000) and Pajak et al. (2000) report the following general trends in their data:

1. TCE concentrations in plant tissues at Hill AFB were 10 to 100 times
    greater than those at CCAS.

2. Metabolite concentrations (2,2,2-trichloroethanol, 2,2,2-trichloroacetic acid,

2,2-dichloroacetic acid, also referred to as TCEt, TCAA, and DCAA) in
plant tissues at HAFB were similar to CCAS. TCEt was the prevalent
metabolite at Hill AFB but not as CCAS.

3. TCE concentrations were higher than metabolite concentrations at Hill AFB;
    however, at CCAS TCE concentrations were less than metabolite
    concentrations.

4. No significant TCE was found in the transpiration gas collected at CCAS.
    However, some TCE was found in transpiration gas collected at Hill AFB.

In an ongoing project at Utah State University, phytovolatilization of TCE (also at
Hill AFB Site OU4) is being characterized. By using a modification of the field sampling
chamber used by Doucette et al. (1998), successful attempts have been made to capture
and measure TCE transpiring from the leaves of some of the same trees being
investigated in this thesis (N. Zaugg, personal communication, June 2001). Preliminary
results from this study strongly suggest that it is possible to both observe and quantify the
transpiration of TCE in field conditions. Further work is planned for spring/summer
2001 to investigate the transpiration of TCE and its relationship to transpired water,
atmospheric temperature, humidity, and solar radiation intensity.

Tree Core Sampling

An article written by Cunningham et al. (1996) stated, "We have redefined plants as
solar-driven pumping and filtering systems." Most of the research has focused on the
treatment potential of vegetation. However, vegetation (specifically trees) can be used as
an efficient and inexpensive sampling tool during the evaluation of shallow,

contaminated aquifer plumes. Only one other group was found during recent literature review to be evaluating this new, potentially beneficial use for vegetation (Vroblesky et al., 1999a).

Studies by Vroblesky and Yanosky (Vroblesky, 1990, 1998; Vroblesky et al., 1999a, 1999b, 1992; Vroblesky and Yanosky, 1990; Yanosky and Vroblesky, 1992, 1995) of the U.S. Geological Survey (USGS) Water Resource Division have found signatures of groundwater contamination recorded in trees growing above the contamination. One such study conducted in 1999 at a forested flood plain near the Savannah River bordering Georgia and South Carolina (Vroblesky et al., 1999a) may be the first to evaluate the use of tree-core analysis as a novel way to quickly and inexpensively map shallow groundwater contamination plumes.

Analysis of tree cores from loblolly pine, sweet gum, bald cypress, oak, and tupelo (located above a shallow aquifer contaminated with 1,2-dichloroethene and TCE) revealed the presence of these contaminants in the trees as well. Vroblesky et al. (1999a) concluded that there might be species variations for tree-core analysis results, vertical variations (within the same tree, but only one tree sampled) for tree-core analysis results, and relative correlations between tree-core analysis results and the groundwater contaminant concentrations. However, a field study by Pajak et al. (2000) completed at Hill AFB during 1999 did not support Vroblesky's findings that tree-core concentrations varied with height.

Currently, the U.S. Air Force Aeronautical Systems Center Environmental, Safety and Health Division is working with the USGS to investigate two separate government owned/contractor operated (GOCO) facilities in Ft. Worth, TX and near Denver, CO.

Coauthored by Vroblesky (USGS) and G. Harvey (U.S. Air Force), these investigations were presented at the Pacific Environmental Restoration Conference in Honolulu, HI from 4-7 April 2000 (G. Harvey, personal communication, May 2000). Both GOCO facilities have groundwater contaminated with TCE, DCE, and vinyl chloride. The Texas site investigation involved the coring of 23 trees comprising 11 different species (among which were eastern cottonwood, oak, and willow) at Carswell Air Force Base's golf course during the month of September. An 80-ft cottonwood tree was also sampled at various heights up its trunk during September. All other cores were taken approximately 5 ft from the ground. The Colorado investigation involved the coring of 14 trees from three different species (eastern cottonwood, gambel oak, and a willow species) collected during the months of May and July at Air Force Plant PJKS (G. Harvey, personal communication, May 2000). Cores were 1.5 inches long and were immediately placed in 20-mL glass vials (sealed with crimped gas tight seal). Analysis involved heating the vials to 40°C for 12 h (reason for 12 h is unknown), cooling to ambient room temperature, followed by headspace analysis (gas sample size of 100 µL) by gas chromatography. Results suggest that tree-core analysis results do follow trends similar to the groundwater concentrations. Consequently, tree-core analysis can be used to map the extent of shallow groundwater contamination. Assuming that the TCE levels within a tree-core are directly affected by changes in transpiration (based on the TSCF), additional sources of error were identified by the Colorado study. Diurnal variation in sap flow rates was highly correlated with climatic variation (strongest relationship was between hourly sap flow and solar radiation). In addition, the relationship between tree size

(diameter) and sap flow was statistically significant (G. Harvey, personal communication, May 2000).

Using Stable Isotopes to Determine Water Sources

In 1932 Urey et al. (1932) reported the discovery of a stable isotope of hydrogen that contained a mass twice that of hydrogen. Curiosity then led additional researchers to question how abundant this stable isotope is relative to the abundance of hydrogen. Between the time of this discovery and the early 1940s, Nier and co-workers (Nier, 1936, 1940, 1990) developed the breakthrough technology enabling the precise measurement of the fractions of light and heavy isotopic forms. Relative abundance is measured using the D/H ratio and ultimately the δD. The delta D or δD is defined as:

$$\delta D = \left[\frac{(D/H)_{sample}}{(D/H)_{standard}} - 1 \right] x\ 1000 \tag{1}$$

The use of δD has revolutionized research in many scientific specialties including physics, atmospheric science, geology and geochemistry, and the biological sciences. Recent advances have resulted in the ability to use δD variations to evaluate plant-water relationships, where the primary hydrogen source is water (Dawson, 1993).

Stable isotopes of hydrogen and oxygen (^{2}H and ^{18}O) in water available for plant use can be measured. Once the ^{2}H and ^{18}O fractions (relative to H and O) have been determined, the resulting unique values can be used to "fingerprint" each available water source. Collection of xylem water from a tree can be used to measure the relative abundance of ^{2}H and ^{18}O within the xylem water. A comparison of stable isotope fractions from both the source water "fingerprints" and the xylem water can be used to

determine which source waters (or fraction of) are being used by the tree for growth.

When planning core sampling for contaminants, it is important to know if the tree is using groundwater during the sampling period. Knowing the season of greatest groundwater use will aid in the detection of contaminants of concern within the tree core. Assuming significant TCE removal occurs as a result of translocation through the tree, one could "scale-up" the expected degradation rate of TCE in a single tree to the degradation expected from a grove of trees. Furthermore, groundwater source information obtained by stable isotope measurements can help to determine the cause of any measured TCE flux within the vegetation. For example, if both the groundwater source and groundwater TCE concentrations have been constant while TCE within the tissue has not, other mechanisms (e.g., TCE metabolism or phytovolatilization) must be contributing to the TCE flux.

Brunel et al. (1994) stated that in order to determine water sources of vegetation using stable isotope measurements, a number of assumptions are necessary: (1) there is no fractionation of water when it is extracted by the roots; (2) there is no significant change in the isotopic composition of sap water within the plant except in the vicinity of the leaf; (3) no significant errors are associated with the sampling of isotopes or in the extraction and analysis of water from plants and soil; (4) the isotopic composition of the soil water is laterally homogenous within the rooting area; (5) the time of sampling is such that time delays associated with transport of isotopes up the plant are not important. Several studies have shown in laboratory and glasshouse experiments that fractionation does not occur in the root uptake of water (Thorburn et al., 1993; Walker and Richardson, 1991). In addition, field validation of this assumption was completed (Brunel et al.,

1994). However, only one exception (where fractionation did occur) has been found in current literature. That exception was demonstrated for a mangrove (Brunel et al., 1997). Validation of the most commonly used sampling and analysis methods has shown that the total error involved in sampling (soil profile and plant canopy heterogeneity), extraction (from soil and plants), and analysis is subject to randomly distributed error of 5% in ^{2}H isotopic composition. If natural variations of δD are greater than this error, stable isotope measurements may be useful. Assumptions 4 and 5 can be verified by field measurements and adequate experimental design.

Fractionation (or partitioning) of D and H among different water sources is primarily due to the differences in their atomic masses. In the environment, fractionation is primarily due to transport processes and phase transitions like evaporation and condensation. For example, as water evaporates it becomes deuterium enriched because hydrogen evaporates at a faster rate than ^{2}H. The different evaporation rates are due to their differing atomic masses. In biological systems, fractionation can be caused by phase transitions, metabolism, chemical reactions, and differential incorporation of D and H due to their different masses and vapor pressures. Temperature and the hydrologic cycle create large variances in the hydrogen isotope ratio (δD) among different source waters. Following the assumptions listed in the previous paragraph, the δD fraction measured within a tree's xylem water would reflect the sum of all δD fractions from all source waters. Consequently, specific mixing models can be used to calculate the individual water source contributions (e.g., groundwater and infiltrated surface water) to the total volume of water used by a tree (Dawson, 1993). Information obtained from a water source study may be used to explain differences in tree-tissue concentrations when

both trees are growing above similar groundwater contamination zones. For instance, a

tree using TCE-contaminated groundwater as its primary source may have higher tree-

tissue concentrations than a tree growing above a similar contaminated groundwater site

where frequent rains provide a more readily available uncontaminated surface water

source.

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CHAPTER 3

SAMPLING AND ANALYSIS METHOD DEVELOPMENT

Abstract

Phytoremediation is an emerging technology promoted as an effective and inexpensive cleanup method for shallow groundwater and soil contaminated with chlorinated solvents like trichloroethylene (TCE). Even as phytoremediation is applied at many TCE-contaminated sites, quantifying the magnitude of uptake has proven to be difficult, especially in a field setting. Consequently, the development and testing of a quick and quantitative collection and headspace analysis (HSA) method for TCE in tree tissue was developed and tested at Hill Air Force Base Operable Unit 4 (OU-4), UT. Because of the mature cottonwood, russian olive, box elder, poplar, and apple trees growing above the shallow TCE-contaminated groundwater aquifer, site OU-4 is an excellent location for studies involving plant uptake of groundwater contaminants. HSA using gas chromatography and electron capture detection was found to be a very quick and relatively easy method that can be used to reasonably estimate TCE concentrations within tree tissue. Collection methods (cordless drills and incremental borers), extraction methods (methanol and pH 2/10 matrix modifying solutions), analysis methods (purge and trap, electron capture detection, mass spectroscopy), and field sampling protocols were evaluated. Seasonal variation, species differences, and variations with respect to height and radial location on a trunk were all examined. Collection of tree cores for comparison of tree-tissue TCE concentrations among trees should involve an

approach that limits variability by collecting from the same height, on the same day (ideally, within the same few hours), from trees of similar diameters, and where possible, from the same species of tree.

Introduction

To evaluate TCE concentrations within the tissue of trees growing above a shallow, contaminated aquifer plume, a quick and quantitative collection and analysis method was developed at Hill Air Force Base Operable Unit 4 (OU-4), UT. Much research has focused on the treatment potential of vegetation. However, vegetation (specifically trees) might also be used as an efficient and inexpensive sampling tool. Vroblesky et al. (1999) summarized research involving the collection of tree tissues for chemical analysis. Completed at a forested flood plain near the Savannah River bordering Georgia and South Carolina, this study may be the first to evaluate the use of tree-core analysis as a novel way to quickly and inexpensively map shallow groundwater contamination plumes.

HSA of tree cores collected using an incremental borer from loblolly pine, sweet gum, bald cypress, oak, and tupelo (located above a shallow aquifer contaminated with 1,2-dichloroethene and TCE) revealed the presence of these contaminants in the trees as well. However, the HSA method used provided only a qualitative evaluation of the presence and general magnitude of TCE within the plant tissue. Others (Doucette et al., 1998, 2000; Pajak et al., 2000; Vroblesky et al., 1999) have also used HSA and have shown that it may be a suitable method for the rapid analysis of tree-core and leaf samples. However, the procedure's effectiveness and reproducibility have not been

adequately evaluated or have not been published to date. As more research seeks to investigate the possibilities of phytoremediation, there is a growing need for quick quantitative methods for sampling and analysis of plant tissues. This study investigates tree-tissue collection methods (coring, drilling), extraction (methanol, adjusted pH sodium chloride saturated water), and analysis methods (headspace, purge and trap, gas chromatography using both electron capture detectors and mass spectrometry) for easy quantification of TCE in tree-tissue samples. Most of the trees were sampled monthly from January 2000 to June 2001 to determine if seasonal variations effect results.

Several aspects of sample collection were evaluated. TCE concentrations in tree tissue obtained using an incremental borer were compared to tree tissues obtained using a cordless drill [a technique used previously at Cape Canaveral Air Station (W.J. Doucette, Associate Professor, Utah State University, personal interview, June 2001)]. Height above the ground surface and radial location were varied to determine any TCE concentration variability resulting from site selection on a trunk. Optimum sampling times were to be determined by evaluating the monthly results. A diurnal study was also completed to evaluate daily tree-core TCE concentrations trends. Following collection, HSA results were compared with purge and trap analyses. Purge and trap analysis (EPA Method 5030B, a more widely used direct analysis method for volatiles) may be more sensitive because of its ability concentrate a sample. Headspace is both quicker and easier than purge and trap. However, HSA is an indirect measurement technique, whereas purge and trap is a direct measurement technique. The methanol extraction used in the purge and trap technique is a rigorous TCE removal process and is very effective at minimizing the volatilization loss of TCE. HSA can be prone to TCE volatilization

losses (especially at elevated temperatures in the headspace analyzer) if the cap is not properly sealed.

Laboratories have often used EPA's SW-846 Method 5021 for HSA of volatiles in soils followed by gas chromatography (GC, involves the use of vials with crimp-on caps and a pH 2, saturated sodium chloride solution). This method, only slightly modified for tree tissue instead of soil, when combined with an electron capture detector (GC/ECD), has shown potential for the quick and quantitative evaluation of TCE and similar compounds in plant tissue samples. Resulting detection of TCE mass is normalized by the tissue dry weight (making comparison among trees or investigators more readily comparable) and reported on a mg/kg basis.

Because TCE is highly soluble in methanol, EPA Method 5030B was used to obtain a direct measurement of TCE in the tissues. Methanol extraction followed by purge/trap analysis using GC/ECD was used to benchmark the actual concentration of TCE within the tree tissue. GC/MS was used to validate the GC/ECD data. Comparing headspace and purge/trap analyses provided a measure of the effectiveness of headspace technique in the quantitative measurement of TCE in tree tissue. Although purge/trap methods are reliable, they are tedious and not very time efficient. HSA involves the collection of a sample, placement in a vial, then placement on an autosampler for analysis--very swift and easy.

Only a few published reports have been found that aid in the development of a tree-tissue collection and HSA protocol. Vroblesky et al. (1999) reported a change in TCE concentration with height above ground level. However, another field study by Pajak et al. (2000) completed at Hill AFB during 1999 did not support the same

conclusion. Currently, the U.S. Air Force Aeronautical Systems Center Environmental, Safety and Health Division is working with the USGS to investigate two separate government owned/contractor operated (GOCO) facilities in Ft. Worth, TX and near Denver, CO (G. Harvey, personal communication, May 2000). Both GOCO facilities have groundwater contaminated with TCE, DCE, and vinyl chloride. The Texas site investigation involved the coring of 23 trees comprising 11 different species (among which were eastern cottonwood, oak, and willow) at the Carswell AFB golf course. An 80-ft cottonwood tree was examined at various heights up its trunk. All other cores were taken approximately 5 ft from the ground. The Colorado investigation involved the coring of 14 trees from three different species (eastern cottonwood, gambel oak, and a willow species) collected during the months of May and July at Air Force Plant PJKS. Cores were 1.5 inches long and were immediately placed in 20-mL glass vials (sealed with crimped gas tight seal). Analysis involved heating the vials to 40°C for 12 h, cooling to ambient room temperature, followed by HSA (gas sample size of 100 µL) by GC.

Assuming that the TCE levels within a tree core are directly effected by changes in transpiration (based on the TSCF), additional sources of error were identified by the Colorado study. Diurnal variation in sap flow rates were highly correlated with climatic variation (strongest relationship was between hourly sap flow and solar radiation). In addition, the relationship between tree size (diameter) and sap flow was statistically significant (G. Harvey, personal communication, May 2000).

Materials and Methods

Tree Core Collection

Tree coring was accomplished using an incremental borer (Forestry Suppliers, Inc.) with a 0.2-inch inner diameter (Fig. 3-1). The incremental borer consists of three parts: a handle, a borer bit (Teflon® coated), and an extractor. The borer bit and extractor fit inside the handle, forming a compact, easily transportable unit. Using the incremental borer, cores approximately 10 cm in length were collected (using a reference mark on the bit) from pre-selected trees growing above a shallow groundwater aquifer contaminated with TCE. Each core was 0.2 inches in diameter (based on the corer bit size).

Height above the ground was generally between 90 cm and 150 cm. Tree structure (obtrusive limbs, thick bark, and compression tissue) and the growing number of existing bore holes placed limitations on the consistency of sample collection at the
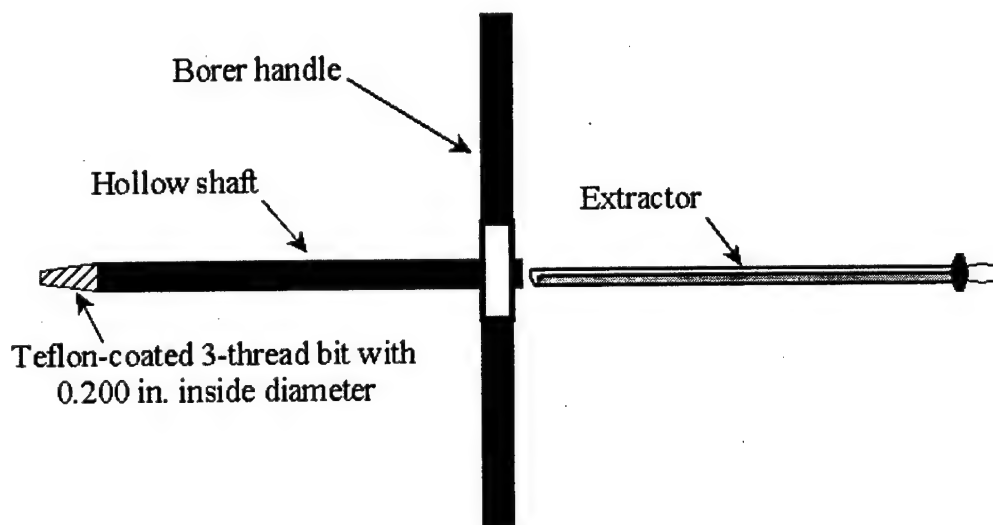
Fig. 3-1. Schematic of an incremental borer used to collect tree cores.

same height above ground level. Initially, the collection of triplicates from any single tree was taken at three points 120° apart around the circumference of the tree. However, Vroblesky et al. (1999) reported concentration differences ranging from 44 to 92% for TCE in samples collected from different sides of the same tree. For comparison, triplicate samples (3 to 5 cm apart) were collected on three sides of a large poplar (60 cm diameter) each 120° apart from the other.

Plant tissue samples (approximately 2 g fresh mass) were removed from the extractor by gloved hand, broken into several smaller pieces, and placed directly into pre-weighed 20-mL headspace vials (Kimble Glass Inc., NJ) containing 10 mL of an acidified (pH 2) saturated sodium chloride solution (referred to as matrix modifying solution in EPA Method 5021). Headspace vials were then sealed using crimped gas tight caps with silicon rubber/ PTFE-lined septa. The extractor and bit were triple rinsed between trees with a soap water solution (Liquinox/deionized water) to remove adhered particulate matter, then rinsed with methanol (ACS grade), then a final rinse using deionized water (listed in order of use). The last rinse of any day was a methanol rinse to prevent rust formation within the handle once the bit and extractor were stored.

The vials were placed on ice and returned to the Utah Water Research Laboratory for analysis. Vials were reweighed upon arrival at the laboratory to determine the actual fresh tissue mass collected and then placed into cold storage at 4° C until needed for analysis. Sample collection for HSA followed closely the procedure outlined in EPA's SW-846 Method 5021 for HSA of soils. Some samples were spiked with a known TCE mass while in the field; however, recoveries were so low that further spiking was halted (tissue sorption being the culprit).

Some samples were also collected using a cordless drill, a 3/16-inch bit, and an aluminum collection apparatus for capturing the drillings (all equipment was decontaminated between trees). The incremental borer is both quick and easy to use but no match for the drill in these two categories. Analysis results were compared between samples collected using the drill and those collected using the incremental borer to determine which collection method recovered the greater amount of TCE within the core.

Sample Preservation and Extraction

In the EPA's SW-846 Method 5021 for HSA of soils, a matrix modifying solution is added to each soil sample to preserve the sample. In the same way, tree cores were collected and placed into headspace vials with 10 mL of matrix modifying solution. The low pH of 2 within the matrix modifying solution serves to preserve the sample. Furthermore, the solute concentration within the water increases the Henry's Law constant (K_h) increasing the sensitivity of the HSA method.

Considering a high pH saturated salt solution might lyse the tissue cells and increase the extraction of TCE from the tissue, a pH 10 sodium chloride saturated matrix modifying solution was made using potassium hydroxide to raise the pH. This pH 10 matrix modifying solution was placed in a side-by-side comparison study with the pH 2 matrix modifying solution. Tree-core analyses were carried out in the same manner for both pH 2 and pH 10 matrix modifying solutions. Sample pairs were collected within 3 to 5 cm of each other to minimize TCE concentration variance between comparison cores. End results in mg TCE per kg dry weight of tissue (mg/kg) were then compared to determine which matrix modifying solution would extract more TCE from the cores.

Some HSA work has been published involving a slightly different method. Vroblesky et al. (1999) reported the placement and analysis of tissue samples (also collected with an incremental borer) in empty glass vials. To replicate this technique, some cores were also placed into headspace vials containing no modifier and results were compared to the pH 2 and pH 10 matrix modifier options.

Methanol was expected to be a more efficient extractant of TCE from the cores than the matrix modifying solution. A simple experiment involving the collection of replicate tree cores was made in order to compare the sensitivity of the two extraction methods. Some tree cores were placed in 40-mL volatile organic analyses bottles (VOAs) with purge and trap (PT) grade methanol (Aldrich, Milwaukee, WI). PT grade water was prepared by purging de-ionized water with nitrogen for approximately 4 to 6 h. Aliquots ranging from 50 to 250 µL were taken from the methanol-extracted core VOAs and spiked into a known volume of PT grade water for analyses. Methanol extraction followed by purge/trap analysis using GC/ECD was used to benchmark the actual concentration of TCE within the tree tissue. GC/MS was used for comparison with all the GC/ECD and GC/headspace data.

Sample Analysis

In the static HSA process, sealed 20-mL vials containing the tissue samples (usually in a solvent) are heated for a predetermined amount of time. The TCE (and other volatiles) will diffuse into and out of the vial's atmosphere (headspace) until a state of equilibrium is reached between the solvent and the headspace. At elevated temperatures and high solute concentrations, the equilibrium between headspace and

Table 3-1. Parameters for the Tekmar 7000 headspace analyzer as used in this experiment.

Parameter	Value	Parameter	Value
Platen temperature	50° C	Inject time	2.00 min
Platen equilibration	0 min	Sample Loop	180° C
Sample equilibration	50 min	Line temperature	180° C
Vial size	20-mL	Injections per vial	1 injection
Mix time	5 min	GC cycle time†	Varied
Mix power (Range 1-10)	8	Method optimization	Off
Stabilization time	1.00 min	Sample size	10 mL
Pressurization time	0.20 min	Static vial pressure‡	17 psi
Pressure equilibration	0.08 min	Loop size	100 µl or 1 mL
Loop fill time	0.40 min	Needle flow	130 mL/min
Loop equilibration time	0.08 min		

† GC cycle time varied with GC temperature program (12 - 20 minutes)
‡ Static vial pressure was set 3 psi above ambient (at 50° C) headspace pressure.

for samples whose aqueous TCE concentrations were expected to range from 1 µg/L up to and above 1 mg/L. The Tekmar 7000 passively fills the sample loops by raising the headspace pressure above its ambient pressure (ambient ~14 psi at 50° C) using nitrogen gas and then using the pressure gradient to fill the sample loop. Nitrogen gas is also used to transport the sample from the headspace analyzer to the GC equipment.

Looking for good sensitivity and a short cycle time, headspace equilibration temperature and time were chosen after a preliminary experiment involving external standards of TCE in 10 mL of matrix modifying solution. These standards were placed in the Tekmar 7000 at five temperatures (ranging from 25 to 65° C) and equilibrated for times ranging from 20 to 120 min. Temperatures were kept below 100°C to avoid the damaging effects of water within the column. A plot of the GC/ECD area count results against the equilibration time helped to determine the best temperature/time combination. The parameters for the Tekmar 7000 are included in Table 3-1.

The Shimadzu GC-14A was operated in the splitless mode using 12 mL/min column flow of lab grade gaseous nitrogen provided by the Tekmar 7000 headspace analyzer. DB-VRX columns (Agilent Technologies, Palo Alto, CA) were used in both 30-m and 75-m lengths. The DB-VRX is a 0.45-mm inner diameter column containing a 2.55 μm coating specially designed for volatile organic analyses. The ECD was normally operated using a range setting of 10 and current setting of 0.5 μV; however, during the course of the experiments the current setting was lowered to maintain ECD sensitivity.

Purge flow through the ECD cell was 2 mL/min and detector temperature was set to 300°C. Make-up nitrogen was used to adjust the total flow (column carrier, purge, and make-up) to achieve a 50 mL/min flow rate through the detector cell. Make-up and purge flow rates were set using a Shimadzu CFC-14PM flow controller. TCE stock standards were made gravimetrically in capillary GC grade methanol (Aldrich, Milwaukee, WI) using stabilized ACS grade TCE (Mallinckrodt Baker, Inc., Phillipsburg, NJ). External standards were made in 10 mL of matrix modifying solution for each analytical run. Matrix modifying solution blanks and calibration check standards were also included in each analytical run.

Two GC temperature programs were used depending on the length of the DB-VRX column. For the 30-m column the following program was used: 60° C for 5 min, then ramping at 40° C/min to 220° C, then holding for 1 min. For the 60-m column the following program was used: 100° C for 8 min, then ramping at 40° C/min to 220° C, then holding for 1 min. GC injection temperature was set to 200°C. No interfering peaks were noted during any of the analysis runs. Class VP data acquisition software (Shimadzu, Columbia, MD) was used to obtain and analyze the ECD data. Method

Table 3-2. Dynatrap™ operational settings used for purge and trap analyses of methanol-extracted tree cores.

PT GC/MS was used	Value	Parameter	Value
Pre-purge	0 min	Valve oven	150° C
Water preheat temp	30° C	GC start	Start
Preheat stir	Disabled	Data start	Start
Preheat time	0 min	GC ready	Open
Purge time	11 min	Sampler cycle time	0 min
Dry purge	2 min	Water standard	No
Desorb preheat	50° C	Water sample volume	10 mL
Desorb temp	180° C	Stir time	0 min
Desorb time	4 min	Stir speed	Off
Bake temp	220° C	Settle time	0 min
Bake time	7 min	Flushes	3
Transfer line temp	170° C	Dilution factor	0
Water trap	Disabled		

detection limits were calculated for both headspace analyzer sample loop volumes following USEPA guidelines (U.S. Code of Federal Regulations, 2001). They were as follows: 100-µL Sample Loop MDL = 2.45 mg/kg, 1-mL Sample Loop MDL = 0.006 mg/kg.

PT GC/ECD and GC/MS were used in comparison studies to evaluate sample solvent options (methanol, pH 2 matrix modifying solution) and to verify the results of the data obtained from the GC/ECD. TCE stock standards were made in PT grade methanol (Aldrich, Milwaukee, WI) using a certified 5,000 µg/mL standard (Supelco, Bellefonte, PA). A Dynatrap™ purge and trap system (Dynatech Precision Sampling Corp., Baton Rouge, LA) was connected to the GC/ECD and provided a column flow of 12 mL/min for the GC/ECD system. Helium was used to purge the sample; nitrogen was used as the carrier gas. Settings specific to the Dynatrap™ are shown in Table 3-2.

PT GC/MS was used for comparison with the PT GC/ECD results. GC/MS equipment included: a Tekmar-Dohrmann 3100 Sample Concentrator (10-mL purge vessel) and Tekmar Precept II autosampler (Tekmar-Dohrmann, Mason OH), HP 6890 Series GC system, a 30-m HP 624 column (0.25-mm inner diameter and 1-μm film thickness), an HP GC autosampler controller, and an HP 5973 Mass Selective Detector. The GC was operated in the split mode using a ratio of 1.6:1 (split flow of 20 mL/min.) and a total flow of 34.7 mL/min. The GC temperature program used was: 40°C - 3 min, 10°C/min to 60°C, 25°C/min to 240°C, hold 1 min. The detector was operated in a selective ion mode for ions 130 and 95 (ion molecular weight/ion charge). HP Enhanced Chemstation Software (all HP products from Agilent Technologies, Palo Alto, CA) linked and controlled the equipment then analyzed the data.

Results and Discussion

Headspace Analyzer Settings

After running a set of experiments to compare different temperature and time settings for the Tekmar 7000, an equilibration time of 50 min was chosen. As can be seen in Fig. 3-2 (data in Table F), the responses from all five temperature ranges were relatively flat after 50 min. Increases in temperature would increase the sensitivity by increasing the Henry's Law constant; however, an increase in temperature also increases headspace pressure that can facilitate a leaky seal. A platen equilibration temperature of 50°C was selected to minimize headspace pressure and the movement of water vapor into the column.

Use of pH 2 or pH 10 Matrix
Modifying Solution

TCE extractions using pH 2 and pH 10 matrix modifying solutions [Tables A.6

through A.9] were not significantly different. Their error bars, in Fig. 3-3, graphically

show that no apparent difference exists between the two different solutions. To remain as

close to the original EPA HSA for volatiles in soils, pH 2 matrix modifying solution will

remain the recommended solution for HSA of tree-tissue TCE concentrations.

Sample Collection Options

When comparing the two separate sample collection techniques (an incremental

borer and a cordless drill), the incremental borer appears to collect a better sample
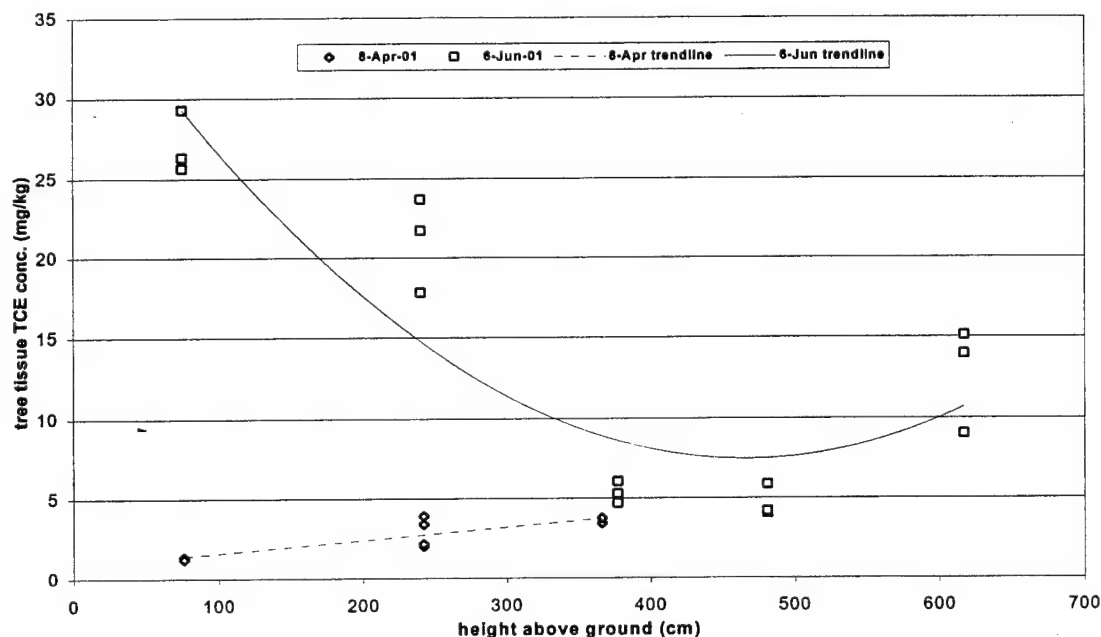
**Fig. 3-2. Optimization of headspace temperature and time settings using 100 μg/L
TCE in modifier solution. Results are averages of duplicates. 50°C – 50 minutes
was chosen.**
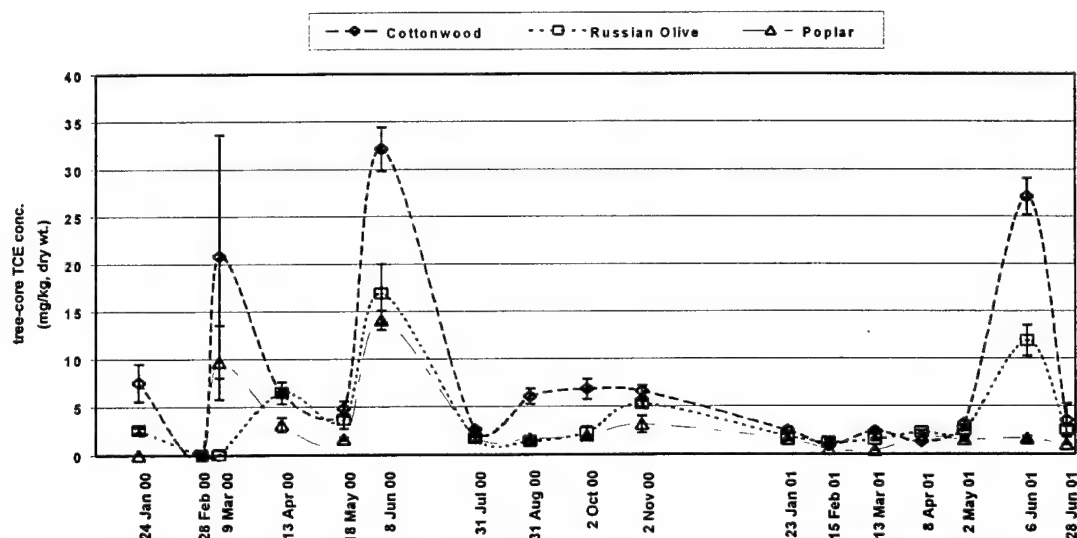
Fig. 3-3. TCE extraction in pH 2 and pH 10 matrix modifying solution using headspace analysis. Columns are averages of triplicates. Error bars denote ±1 standard deviation.

because significant TCE loss was measured [Fig. 3-4, data in Table A.6] when a cordless drill was used. This TCE loss may be related to the increase in surface area for the shavings (promoting evaporation from the surface) and an increase in temperature during sample collection (due to the friction generated with a drill bit).

Purge and Trap vs. Headspace

There is no easy way to quantitatively measure the mass of TCE within a tree core. The use of C-14 labeled TCE would be a good technique for best mass recovery. The best technique would ensure that the TCE moves into and throughout the tissue the same way it would in the natural environment. However, growing a decades old mature tree in radiolabeled water is not feasible. During this experiment, an assumption was made that methanol extraction of TCE from tree tissue using a purge and trap analysis method was the most rigorous method available. Being a direct extraction method, purge
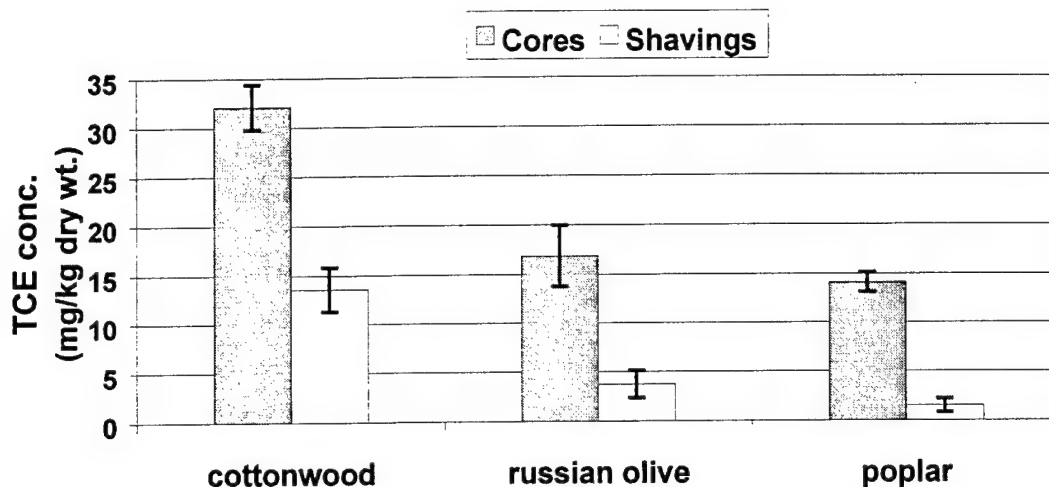
Fig. 3-4. Tree-tissue TCE concentration comparison between drilling and coring collection methods. Error bars denote ± 1 standard deviation among triplicate samples.

and trap (PT) was assumed to remove the majority of the TCE. PT results were used as a benchmark for comparison with HSA results. Fig. 3-5 shows that PT analyses did recover more of the TCE from the cores [data in Tables A.22, A.23, B.1, and B.2] than HSA. Electron capture detection (ECD) was used for both the headspace and PT analyses methods. Purge and trap using mass spectroscopy (MS) was used as a qualitative check for the ECD results.

A general relationship was found between purge and trap and HSA. The regression shown in Fig. 3-5 shows that PT/ECD consistently measured more TCE in the tree cores. By comparing April 2001 HSA results [Tables A.22, A.23] with April 2001 PT/ECD results (Table B.2), PT/ECD detected 2.7 times more TCE (with 31% standard deviation of the mean). Since HSA are an indirect method, an increase using PT was expected. Using this regression, an estimate of actual TCE present in a tree core can be made using the indirect method of HSA.

Fig. 3-5 only includes data collected for those trees growing above TCE-

contaminated groundwater ranging from 800 to greater than 4000 µg/L. Additional data were collected for tree tissue obtained from trees growing above groundwater contaminated with only trace amounts of TCE (at or below the method detection limit of 0.1 µg/L as reported by Hill AFB Environmental Management Directorate). PT/ECD could not detect TCE in the cores at these sites. Purge and trap MS did detect TCE in the same methanol extract. PT/MS results were approximately 55 times greater than the headspace ECD values. It is not known why the PT/MS results were so much greater than the PT/ECD values for cores collected at the site of low groundwater TCE contamination. Different standards and a much larger headspace sampling loop (1 mL vs. 100 µL) may have resulted in this much larger difference between the PT and HSA results. Although the PT/ECD method appears to detect approximately 2.7 times more
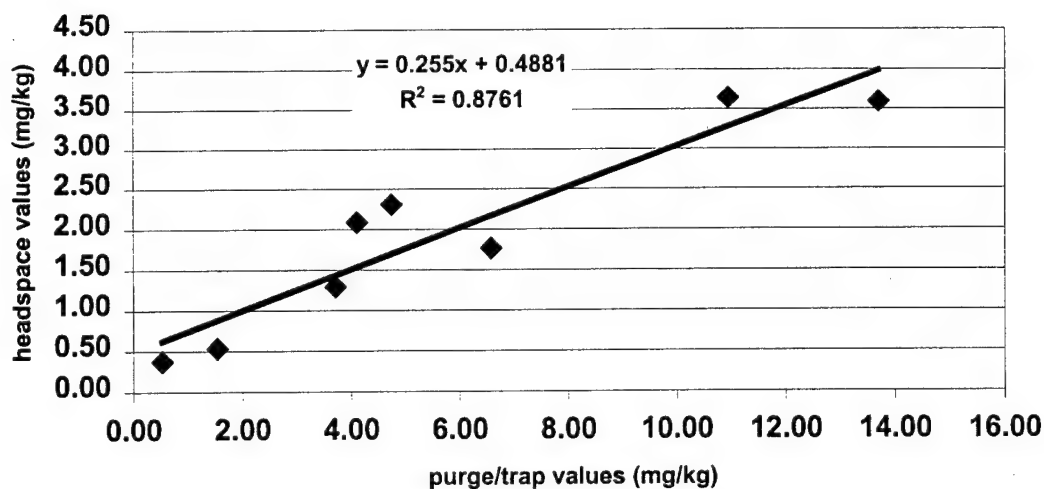
Fig. 3-5. Comparison of headspace and purge/trap analysis (PT) results. All points reported as tree-tissue TCE concentrations (mg/kg dry weight). PT results are averages obtained from duplicate PT electron capture detection and PT mass spectroscopy data.

TCE than the headspace for the medium and high TCE-contaminated groundwater, this
tentative relationship is not valid at the low TCE-contaminated site.

Variance Due to Radial Location

Tree-tissue TCE concentrations were found to vary with radial location around
the trunk [data in Table A.15]. A single poplar tree (59-cm diameter) was sampled using
an incremental borer at three locations 120° apart around the circumference. Each
sampling location was sampled three times (each core 2-4 cm apart) for a total of nine
cores. Analysis of variance returned a P-value of 0.00179 at a 0.01 probability showing
that the three separate means were significantly different. A schematic (see Fig. 3-6)
graphically represents the locations, tree-tissue concentration means, and standard
deviations for each location. Vroblesky et al. (1999) showed concentration differences
from 44 to 92% for samples from different sides of a tree while samples only 25 mm
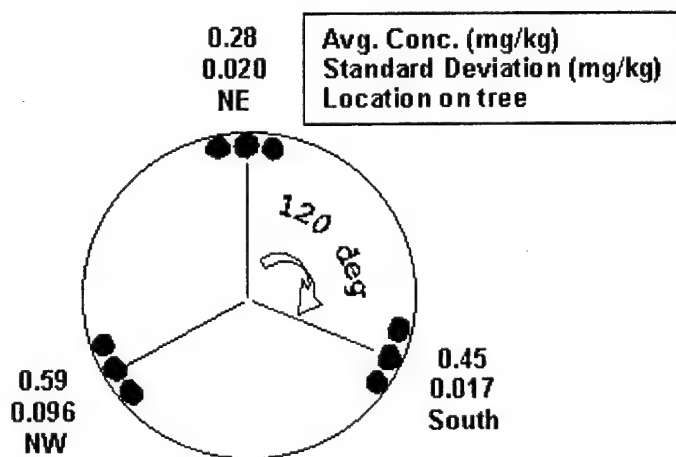
Fig. 3-6. **Three sets of triplicate cores from a poplar (59-cm diameter) collected**
2-4 cm apart.

apart were only 15.5% different. Although this particular finding of radial variance is supported in professional literature, limitations exist for its future use.

Although the data do show significant radial variability, it is not known how predictable this variance may be. Because samples were collected for radial variance measurements from only one tree, at one height, at one point in time, further field level evaluations of other species, sizes, and during other seasons may be necessary before applying this information to other trees or sites.

Concentration Dependence on Tree Height

Field data exist demonstrating a tree-core TCE concentration dependent on height. Vroblesky et al. (1999) reported a decrease in TCE concentration in a bald cypress tree with an increase in trunk height (20 m). Pajak et al. (2000) examined poplar, russian olive, and willow (10 m maximum) at Hill AFB OU-2 and did not identify any
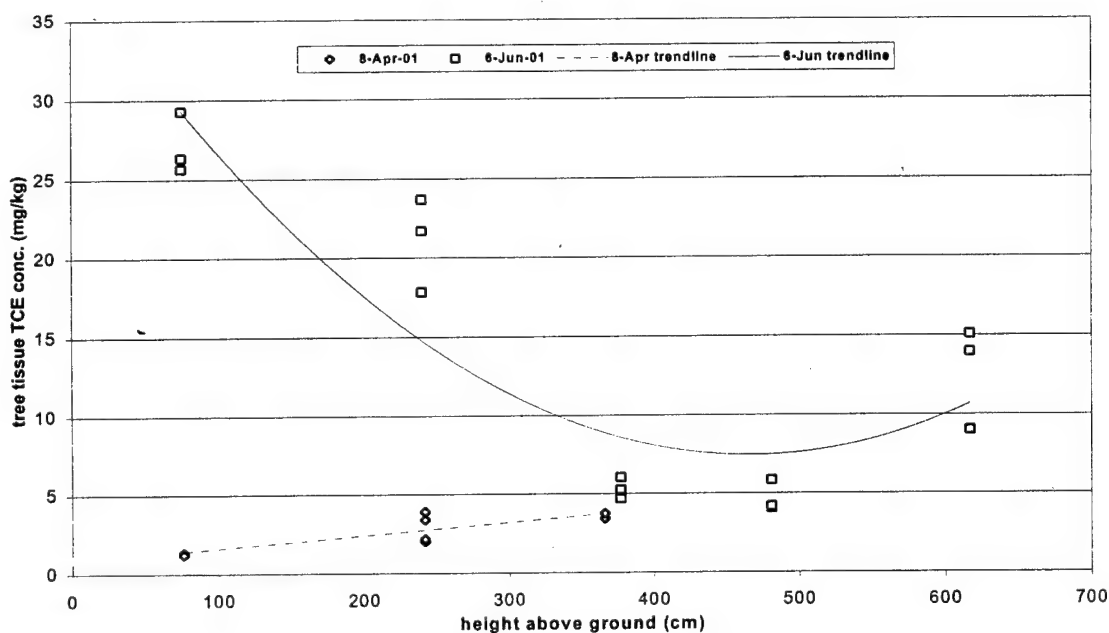
Fig. 3-7. Tree-tissue TCE concentrations measured at different heights on a cottonwood in April and June 2001.

significant concentration trends with respect to sampling height. Fig. 3-7 shows separate

sampling events that involved the collection of tree cores at various heights up a

cottonwood trunk. Generalized concentration trends may be seen within the graph;

however, no trend appears to be consistent with time or with distance above the ground.

Fig. 3-7 shows different trends within the span of two months [data in Tables A.23 and

A.27]. Significant decreases in TCE concentration occurred from 75 cm up to 377 cm.

However, from 481 cm to 617 cm an increase occurs. Since a known variability has been

established dependent on radial location, the switch in sampling location from west to

north to northwest (required because of accessibility limitations) may have skewed the

results.

Daily and Seasonal Variation Effects

To determine if tree-core TCE concentrations vary during a day/night cycle, a

single cottonwood was routinely sampled using an incremental borer from 2045 h one
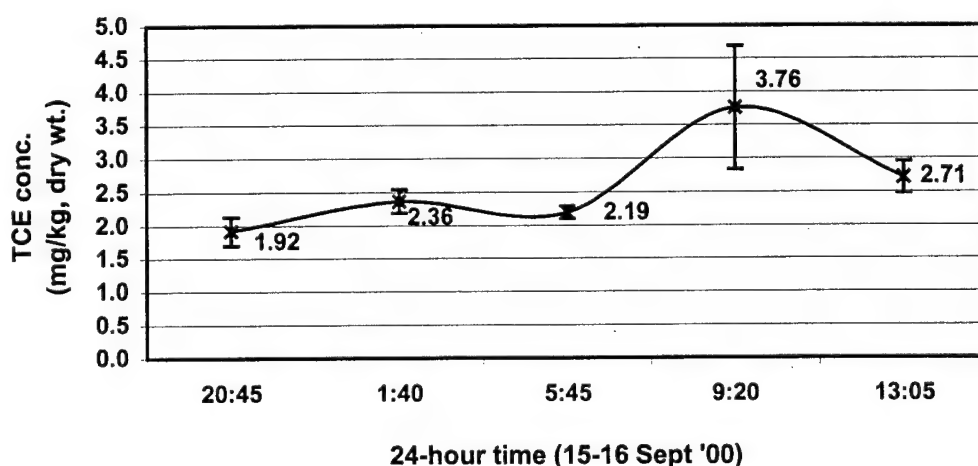
Fig. 3-8. Daily tree-tissue TCE concentrations in a large cottonwood growing above groundwater > 4000 µg/L TCE. Error bars denote ±1 standard deviation.

evening until 1305 h the next afternoon [see Fig. 3-8, data in Table A.11]. Triplicate samples (2-4 cm apart) were collected at each point in time. Single-factor ANOVA analysis using an alpha of 0.05 concluded that mean tree-tissue concentration values (taken from triplicates) were significantly different (P value = 0.00989). At 0920 h a significant increase was measured followed by an apparent decrease at 1305 h. Although the increase might be attributed to an increase in transpiration, error may have been introduced by sample location. Radial location around the trunk was not recorded or controlled. Therefore, no solid conclusions can be made regarding day and night TCE concentrations.

Tree-tissue TCE concentrations appear to fluctuate month to month from January 2000 to June 2001 (see Fig. 3-9). Although some fluctuations were not significant based on their error bars, those values obtained in June were significant. In June of both years, very large increases were observed for the cottonwood and the russian olive. Laboratory studies have generally shown that plant uptake of organic contaminants like TCE is a function of transpiration, the hydrophobicity of the chemical (expressed as the octanol/water partition coefficient, or K_{ow}), and the concentration of the chemical in the water used by the plant (Briggs et al., 1982; Bromilow and Chamberlain, 1995; Burken and Schnoor, 1998; Cunningham et al., 1996; Orchard et al., 2000). Recent work regarding the biophysical mechanisms of TCE uptake concluded that a decrease in seedling water use from summer to winter explained a large seasonal difference in TCE flux (Nietch et al., 1999). Having established that TCE is transported through a tree via the transpiration stream, a retention mechanism is still necessary for tree-tissue TCE concentrations to vary with seasonal transpiration rates. Sorption has been established as
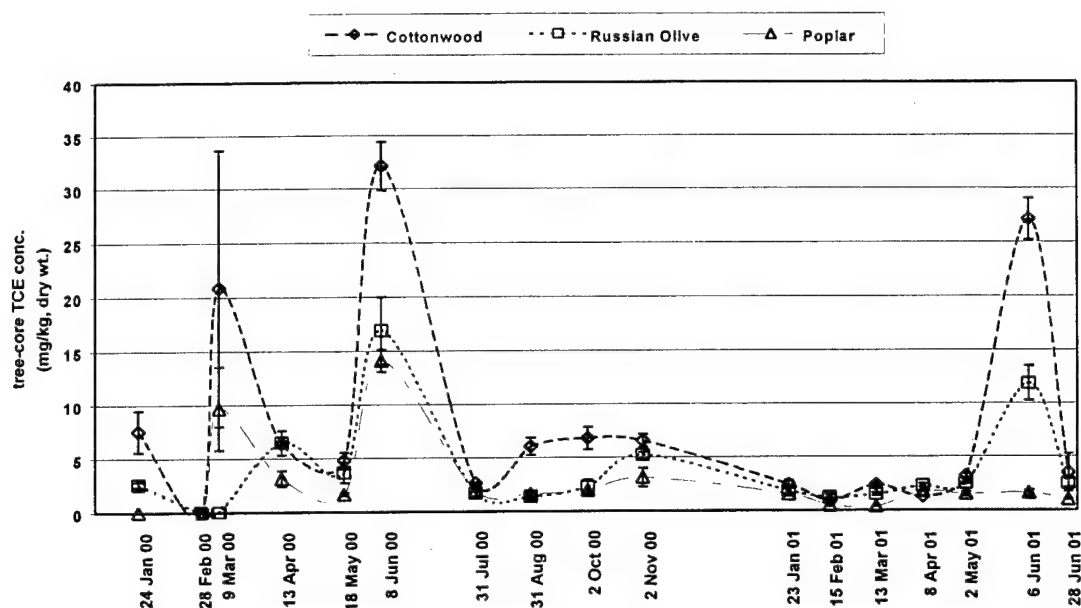
Fig. 3-9. Seasonal trends and species comparisons for TCE concentrations in tree cores. Error bars denote ±1 standard deviation.

a potential mechanism for organic contaminant retention within the tissues of trees

(Mackay and Gschwend, 2000; Trapp et al., 2001). Therefore, tree-tissue TCE

concentrations are expected to fluctuate with the seasons. The high tree-tissue TCE

concentrations shown in Fig. 3-9 for the months of June are aligned with the month of

highest average transpiration rates for this portion of northeast UT, where Hill AFB is

located.

Species Variations

Having established that transpiration rates can affect tree-tissue TCE

concentrations, it is no surprise that species differences might also be noted. Vroblesky

et al. (1999) did report notable species differences between the oaks and the adjacent bald

cypress or loblolly pines. Fig. 3-9 reveals basic trends that may be associated with tree

species (data obtained from Appendix A). Reported tree-tissue concentration values for

the cottonwood were generally above the russian olive and poplar species. Russian olive also appears to be higher than the poplar. Although these trees are within 30 ft of each other, there is the possibility that groundwater concentration could vary enough to effect the measured tree-tissue concentrations. Only one well was sampled from January to June of 2001. Its groundwater TCE concentration ranged from 4375 to 4890 µg/L, fluctuating only ~6% from its mean. Species may also result in different tree sizes. Vroblesky et al. (1999) established a significant relationship between tree diameter and concentration. This is not a surprise since a large diameter tree would need to transpire more. Diameters from cottonwood to russian olive to poplar are: 220 cm, 145 cm, and 147 cm:165 cm (split trunk).

In conclusion, this work has evaluated several interferences to the use of HSA of tree-core tissue as a method for quantifying the actual TCE concentration within a tree. It is recommended that final results be reported with respect to dry weight of plant tissue. This permits others to build relationships that incorporate wood/water partition coefficients as a way to evaluate results across species. The recommended procedure for quick and effective sampling and analysis of tree-tissue cores would include the use of an incremental borer and HSA with pH 2 matrix modifying solution. Sampling in replicates around the trunk's circumference would best identify the cross sectional concentration by averaging any variations with respect to radial location. Tree size may have significant effects on the outcome of tree-tissue TCE concentrations. To examine species differences, trees must be close to one other and have similar trunk sizes. Finally, collection of samples for comparison among trees should involve an approach that limits variability by collecting from the same height, on the same day (ideally within the same

period of the day), from trees of similar diameters, and where possible, from the same

species of tree.

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CHAPTER 4

USING HEADSPACE ANALYSIS TO EVALUATE THE RELATIONSHIP BETWEEN TRICHLOROETHYLENE CONCENTRATIONS IN TREES AND ASSOCIATED GROUNDWATER

Abstract

An 18-month field study was completed at a shallow trichloroethylene (TCE)-contaminated groundwater aquifer at Hill Air Force Base (Ogden, UT) to evaluate the potential use of cottonwood (*Populus deltoides*), russian olive (*Elaeagnaceae elaeagnus*), poplar (*Salicaceae populus*), apple (*Malus pumila*), and box elder (*Acer negundo*) as efficient, inexpensive water sampling tools. Tree-core samples were analyzed using a headspace gas chromatography method. Tree-tissue concentrations ranging from 0.006 to 32.13 mg TCE/kg dry weight of tissue were found in trees growing above TCE-contaminated groundwater (non-detect - 4000 μg/L). Headspace analysis (HSA) using gas chromatography with electron capture detection (GC-ECD) was compared with methanol extraction and purge/trap GC-ECD. Methanol extraction was approximately 2.7 times more effective at removing TCE from tissue for trees growing above groundwater contaminated with 800 μg/L. This multiplication factor may be useful in the conversion of HSA results into estimated total mass of TCE in tree tissue. Contaminated groundwater samples (~0 - > 4000 μg/L TCE) were collected from wells close to the sampled trees for comparison with tree-tissue concentrations. Tree-tissue concentrations were slightly

dependent on radial (2X) and vertical (6X) trunk position. Other sources of potential variance included tree size, species, and environmental factors. Stable isotopes of hydrogen were measured in groundwater, rainwater, and xylem sap to verify the tree's primary water source. Significant and measurable plant uptake of TCE by these trees creates possibilities for their use as efficient, inexpensive water sampling tools.

Introduction

Opportunities may exist for using trees as an efficient and inexpensive water sampling tool for site characterization of shallow groundwater contaminant plumes. Plant uptake of organic groundwater contaminants like TCE is thought to be a passive process by which the contaminants move into the plant through root uptake. Laboratory studies have generally shown that uptake is a function of the amount of water transpired by the plant, the hydrophobicity of the chemical expressed as the octanol/water partition coefficient (K_{ow}), and the concentration of chemical in the water used by the plant (Briggs et al., 1982; Bromilow and Chamberlain, 1995; Burken and Schnoor, 1998; Cunningham et al., 1996; Orchard et al., 2000). This suggests that for a given chemical, the amount of plant uptake in a field setting, and most likely the concentration in the plant, should be a function of the concentration of the chemical in the groundwater in use by the plant. One implication of this possible relationship is that plants could be used as in-place groundwater pumps augmenting (possibly replacing) more costly wells.

While this premise has been tentatively supported in a few field studies, additional data are needed. A study conducted in 1999 at a forested flood plain near the

Savannah River bordering Georgia and South Carolina may have been the first to evaluate the use of tree-core analysis as a novel way to quickly and inexpensively map shallow groundwater contamination plumes (Vroblesky et al., 1999). HSA of tree cores collected using an incremental borer from loblolly pine, sweet gum, bald cypress, oak, and tupelo (located above a shallow aquifer contaminated with 1,2-dichloroethene and TCE) revealed the presence of these contaminants in the trees as well. However, the particular HSA method (cores placed in empty headspace vials) provided only a qualitative evaluation of the presence and general magnitude of TCE within the plant tissue.

Doucette et al. (1998, 2000) and Pajak et al. (2000) have also used HSA and have shown that it may be a suitable method for the rapid analysis of tree-core and leaf samples. Uptake of TCE by trees growing above a shallow contaminated groundwater site at Cape Canaveral Air Station (CCAS) was evaluated using HSA techniques. TCE concentrations in tissues at HAFB were 10 to 100 times greater than those at CCAS even though both sites had similar groundwater contamination (Pajak et al., 2000).

Theorizing that the shallow groundwater at HAFB is a primary water source and shallow groundwater at CCAS is not, initial characterization of HAFB water sources using stables isotopes of hydrogen was completed. Fractionation (or partitioning) of deuterium and hydrogen among different water sources is primarily due to the differences in their atomic masses. In the environment, fractionation is primarily due to transport processes and phase transitions like evaporation and condensation. With one exception for a mangrove (Brunel et al., 1997), several studies have shown in laboratory and

glasshouse experiments that fractionation does not occur in the root uptake of water (Thorburn et al., 1993; Walker and Richardson, 1991). Analysis of tree cores, groundwater, and surface water were completed to gain a better understanding of water sources in use by trees at HAFB. Seeking more definitive data regarding the specific use of tree-tissue HSA for groundwater plume delineation, eight trees growing above shallow TCE-contaminated groundwater were monitored for an 18-month period at Hill Air Force Base in UT.

Hill Air Force Base Operable Unit #4 (HAFB OU-4) is located 25 miles north of Salt Lake City, UT on the northern edge of the Weber Delta, a terrace approximately 300 feet above the surrounding valley floors of Weber and Davis Counties. The steep slopes promote groundwater seepage during wet periods and generally promote shallow depths to the groundwater table. Easy access to this shallow groundwater has promoted the substantial growth of various tree species including apple, willow, russian olive, poplar, box elder, and cottonwood. HAFB lies in a semiarid region with an average annual rainfall of less than 20 inches/year. May is usually the wettest month, and June and July are the driest months. Average evaporation is approximately 45 inches of water per year. Winter snowfall can be significant and provides recharge for the shallow aquifer during the winter months.

Sampling site selection was made after initial review of historical groundwater data for site OU-4 (Haraden, 1999). Groundwater data from January 2001 through June 2001 were provided by HAFB Environmental Management Directorate through their current contract for sampling and analyses. The 2001 data coincide with the historical

data provided by Haraden's USGS report listed above. The groundwater concentrations have remained relatively constant for several years.

A control site (known to be TCE free) was chosen upgradient of the groundwater TCE plume. Three additional sites (A, B, and C) were chosen to bracket ranges of known groundwater TCE contamination at OU-4 as determined by well data (3700 – 5000 µg/L, 800 – 1700 µg/L, and 0 – 1 µg/L). Appendix C contains summary information on the wells. Criteria for tree selection at each site included distance from groundwater wells (the closer the better), availability of different species (the more the better), and tree size (the bigger the better). Table 4-1 contains basic information on the eight trees sampled over an 18-month period from January 2000 to June 2001. The general layout of groundwater contamination and the general location of the trees is shown in Fig. 1-2. Groundwater TCE concentrations may vary between and among the

Table 4-1. Primary trees sampled at Hill AFB Site OU-4.

Identifier	Species	Circumference (cm)	Location (ft, deg from well #)	Groundwater TCE Conc. (µg/L) and Site ID
C-8 †	Russian olive	300	358'315° from U4-037	0 (Control)
C-1	Box elder	104	35'232° from U4-075	0 – 1 (Site C)
C-2	Apple	109	28'210° from U4-075	0 – 1 (Site C)
C-7	Poplar	186	50'9° from U4-064	800 – 1700 (Site B)
C-11	Poplar	155	65'22° from U4-064	800 – 1700 (Site B)
C-3	Cottonwood	270	20'203° from U4-065	3700 – 5000 (Site A)
C-4	Russian olive	145	33'190° from U4-065	3700 – 5000 (Site A)
C-10 ‡	Poplar	264	110'270° from U4-006	3700 – 5000 (Site A)

†- Control tree located up-gradient of groundwater flow and contamination source.
‡- Split trunk – ground level is 264 cm and splits into two trunks of 147 cm and 165 cm.

root structure and would be very difficult to measure. Therefore, an assumption is made that groundwater TCE concentrations in the nearest wells represent the TCE groundwater concentrations in use by the tree.

Materials and Methods

Tree coring was accomplished using an incremental borer with a 0.2-inch inner diameter. Each core was approximately 10 cm in length. Initially, the collection of replicates from any single tree was taken at two or three points around the circumference of the tree. However, Vroblesky et al. (1999) reported concentration differences ranging from 44 to 92% for TCE in samples collected far apart on the same tree. Beginning in May 2000, replicate samples were collected 3 – 5 cm apart to help determine the reproducibility of the collection and analytical methods. Height above the ground was generally between 90 and 150 cm because locations of approximately waist to chest height were easiest to sample using the incremental borer.

These tree-tissue samples (approximately 2 g fresh mass) were removed by gloved hand, broken into smaller pieces, and placed directly into preweighed 22-mL headspace vials (Kimble Glass Inc., NJ) containing 10 mL of an acidified (pH 2) saturated sodium chloride solution (sampling and analyses procedures were taken from EPA SW-846 Method 5021 for HSA of soils). This EPA method incorporates the use of a modifier solution that helps to provide a constant matrix making quantification easier (external standards can be spiked directly into the modifier). In addition, the acidified modifier solution enhances headspace sensitivity by raising the Henry's Law constant (K_h) and preserves the sample preventing biological TCE degradation. Headspace vials

were then sealed using crimped gas tight caps with silicon rubber/PTFE-lined septa and placed into cold storage at 4° C until needed for analysis. Initially, two to three tree-tissue samples were spiked with TCE in the field. Subsequent analysis of these spiked tissue samples resulted in poor TCE recoveries. Spiked matrix samples (TCE + modifier) analyzed concurrent with the spiked tissue samples did not reveal the same matrix effect. Thus, sorption of TCE to the tissue was suspected (and eventually proven and measured in Chapter V). Overall, field spikes were included in the field collection procedures from January to August 2000 (Appendix A, all Tables A.1 through A.11) then halted for the remainder of the research.

The incremental borer and extraction bit were sequentially rinsed between trees with a soap water solution to remove adhered particulates, and then rinsed with methanol and deionized water. Upon return to the laboratory, all samples were reweighed to determine the fresh mass collected. All samples were placed on a horizontal shaker for 24 h prior to analysis to promote partitioning equilibrium between the core, the modifier solution, and the headspace.

A Tekmar 7000 headspace analyzer and 7050 carrousel (Tekmar-Dohrmann, Mason OH) were used to prepare all samples for HSA. Gaseous samples collected by the Tekmar systems (equipped with either a 1-mL or 100-μL sample loop volume) were sent to a Shimadzu GC-14A equipped with an electron capture detector (ECD). The large sample loop was used for samples whose aqueous TCE concentrations were expected to range from 0.1 μg/L to 10 μg/L. Similarly, the small sample loop was used for samples whose aqueous TCE concentrations were expected to range from 1 μg/L up to and above

1 mg/L. Equilibration temperature and time were 50° C and 50 min, respectively. Both 30- and 60-meter DB-VRX columns (Agilent Technologies, Palo Alto CA) were used for the gas chromatography. In an attempt to remove any residual sodium chloride that may have become entrained within the tissue, all tree cores were tumbled for three consecutive days in 40-mL volatile organic analysis vials containing deionized water (changing the water each day). Following this rinsing procedure, all cores were dried in a 103° C oven for 24 h to obtain individual dry weights. Finally, these dry weights were used to report the final results of HSA on a mg TCE per kg dry weight basis (mg/kg). Method detection limits were calculated for both headspace analyzer sample loop volumes following Appendix B to Part 136, 40 CFR Chapter 1 (7-1-89 edition). They were as follows: 100-uL Sample Loop MDL = 2.45 mg/kg, 1-mL Sample Loop MDL = 0.006 mg/kg.

Since there is no easy way to quantitatively measure the mass of TCE within a tree core (except for growing a tree in radiolabeled TCE and combusting the samples), a comparison was made between the use of the modifier solution and methanol extraction (expected to be much more rigorous). EPA Method 5030B, a standard purge and trap method for volatiles, has often been used to analyze for volatiles contained within the methanol extract of soils. Methanol extractions (tree cores in 40-mL vials containing approximately 40 mL of methanol, tumbled for 24 h prior to analyses) were analyzed using gas chromatography by purge and trap analysis (using an ECD and then verified with mass spectroscopy on a different system). For an in-depth description of all analytical methods used, see Chapter 3.

Groundwater data for January 2001 through June 2001 (Appendix C) was provided by Hill AFB Environmental Management through their current contract for sampling and analyses. Groundwater was sampled on the same day as the collection of the tree cores for the months of April, May, and June 2001 (and within one week of core collection for January through March 2001). A historical review of groundwater concentrations measured at the wells used in this study shows that groundwater TCE concentrations have been quite steady since 1996 (Haraden, 1999).

For the source water study at Hill AFB OU-4, tree cores, groundwater, and rainwater were collected. Water samples (ground and rain) were collected by HAFB personnel and kept at or below 4° C in vials with zero headspace vials to prevent isotopic changes due to evaporation. Tree cores were collected, broken into smaller pieces, placed in 7-mL vials, and immediately frozen in the field using dry ice. The cores were kept frozen until the University of Utah's Stable Isotope Ratio Facility for Environmental Research (SIRFER) was ready to extract the water using a special steam distillation process (Ehleringer and Osmond, 1989). The resultant hydrogen gas is consequently analyzed using a gas isotope ratio mass spectrometer for determination of the deuterium/hydrogen ratio and reported in delta notation (‰, per one thousand) relative to standard mean ocean water (SMOW), an accepted standard for deuterium/hydrogen ratios.

Results and Discussion

Headspace Analysis for Quantification
of TCE in Tree Tissue

In Chapter 3 tree-tissue concentrations were shown to be statistically different (varying by 2X) at three locations 120° apart around a tree's circumference (Fig. 3-6). Vroblesky et al. (1999) also observed this radial variance. In order to minimize the variance of replicate tree-tissue samples, all tree-tissue samples collected after May 2000 were collected on the same side of the tree (usually within 3 to 5 cm of each other). In April 2001 [Appendix A Tables A.22, A.23, Appendix B Tables B.1, B.2] four separate tree-tissue samples were collected using the incremental borer (each 3 to 5 cm from the other). One pair of these tree-tissue samples was placed in headspace vials containing modifier solution for eventual GC-ECD analysis. The other pair was placed in amber 40-
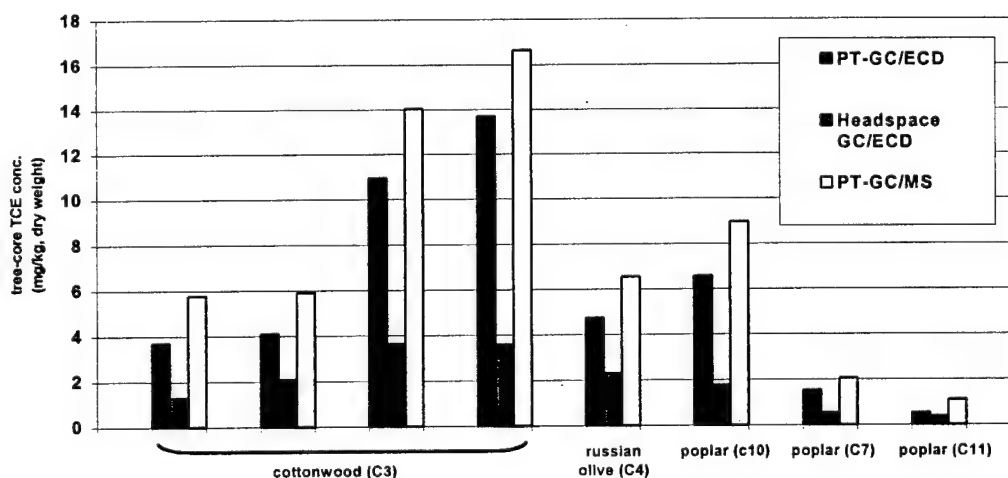
Fig. 4-1. Comparison of purge and trap (PT) gas chromatography (GC) method results [both electron capture detection (ECD) and mass spectroscopy (MS)] with headspace analyses results for trees from sites A and B.

mL volatile organic analyses vials containing approximately 40 mL of methanol. The methanol extract was later analyzed by purge and trap using both GC/ECD and GC/MS. Purge and trap analyses results were similar and predominantly greater than the HSA results (Tables B.1 and B.2). A general relationship was found between purge and trap and HSA. Fig. 3-5 and 4-1 show that PT/ECD consistently measured more TCE within the tree cores. By comparing April 2001 HSA results (Tables A.22, A.23) with April 2001 PT/ECD results (Table B.2), PT/ECD detected 2.7 times more TCE (with 31% standard deviation of the mean). Since HSA is an indirect method, an increase using PT was expected. Using this regression, an estimate of actual TCE present in a tree core can be made using the indirect method of HSA.

Both Fig. 3-5 and 4-1 include only data collected from trees growing above TCE-contaminated groundwater concentrations ranging from 800 to greater than 4000 µg/L. Additional data were collected for tree tissue obtained from trees growing above groundwater contaminated with only trace amounts of TCE (at or below the method detection limit of 0.1 µg/L as reported by Hill AFB Environmental Management Directorate). PT/ECD could not detect TCE in the cores at these sites. Purge and trap MS did detect TCE in the same methanol extract. PT/MS results at the low groundwater TCE concentration site were approximately 55 times greater than the headspace ECD values. It is not known why the PT/MS results were so much greater than the PT/ECD values for cores collected at this site. Different standards and a larger headspace sampling loop (1 mL vs. 100 µL) may have resulted in this increased difference between the PT and HSA results. Although the PT/ECD method appears to detect approximately

2.7 times more TCE than the headspace for the medium and high TCE-contaminated groundwater, this tentative PT/headspace relationship is not valid at the low TCE-contaminated groundwater site.

Groundwater/Tree-Tissue TCE Concentration Relationship

A qualitative relationship between groundwater TCE concentrations and tree-tissue TCE concentrations is evident in Fig. 4-2. Taking the average tree-tissue and groundwater concentrations collected from January to June 2001 (see tables in Appendices A and C), one can see in Fig. 4-2 that as groundwater concentrations increase, so do the tree-tissue concentrations. Results were generally similar among trees at each site with the exception of the June 2001 data (Table A.27). June 2001 was
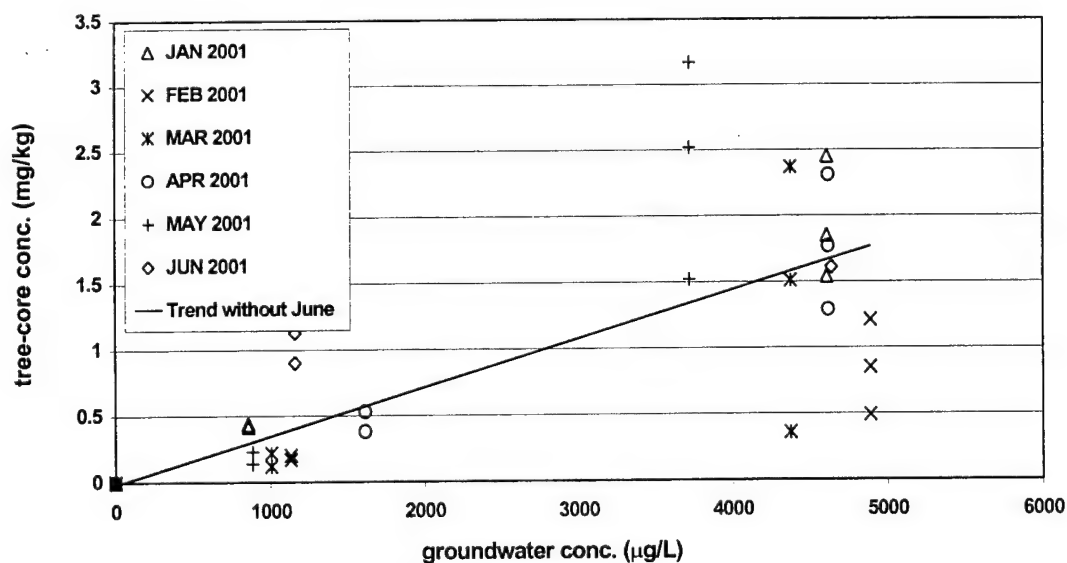
Fig. 4-2. Groundwater TCE concentration plotted against tree-core concentrations. Each data point is an average value for a single sampled tree.

significantly higher than most other months. When plotting a linear regression (used in this case for showing general trends only) as in Fig. 4-2, the June 2001 data strongly skews what would appear to be a strong relationship between groundwater and tree-core TCE concentrations. However, a definitive relationship requires much more data and additional research into the sources of variability (e.g., environment, season, tree size, tree species).

Potential Environmental Factors Affecting
Tree-Tissue TCE Concentrations

If seasonal trends occurred, one would expect those trends to be visible at more than one sampling site. Tree-core TCE concentrations collected in June 2000 at site A (Fig. 4-3) were higher than other months in 2000. However, no June 2000 data were collected at the medium location, site B (data for poplar growing above $800 - 1700$ µg/L TCE). Consequently, a comparison between the high and medium TCE groundwater concentration sites could not be done. Fig. 4-3 and 4-4 both show June 2001 as significantly higher than other months (with exception to March 2000 whose large variance might overshadow any seasonal effects). June 2001 increases might be due to seasonal effects.

Fig. 4-5 (reports TCE concentrations from apple, box elder, and russian olive trees located above groundwater containing < 1 µg/L TCE) did not reveal any appreciable rends. The russian olive in Fig. 4-5 is a control tree located above groundwater consistently reported as at or below the method detection limit (usually 0.2 µg/L using EPA Method SW8260B). Only one sample was taken (no replicates) in May 2000 from
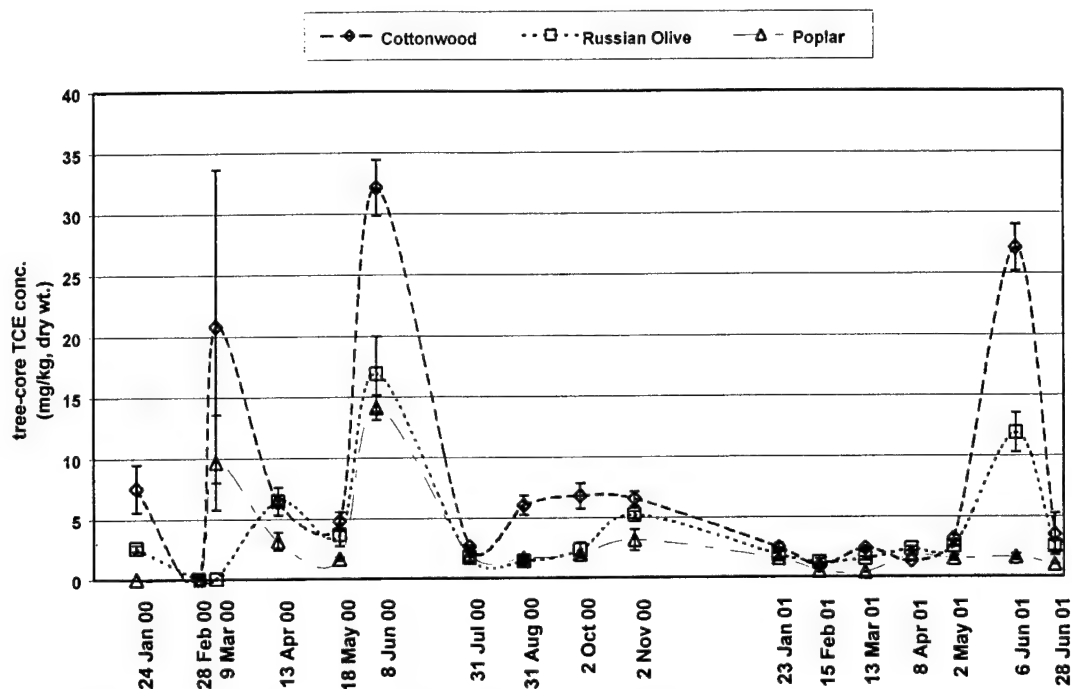
Fig. 4-3. Time series plot of tree-tissue TCE concentrations at site A (high TCE concentrations in groundwater). Error bars denote $\pm$1 standard deviation.
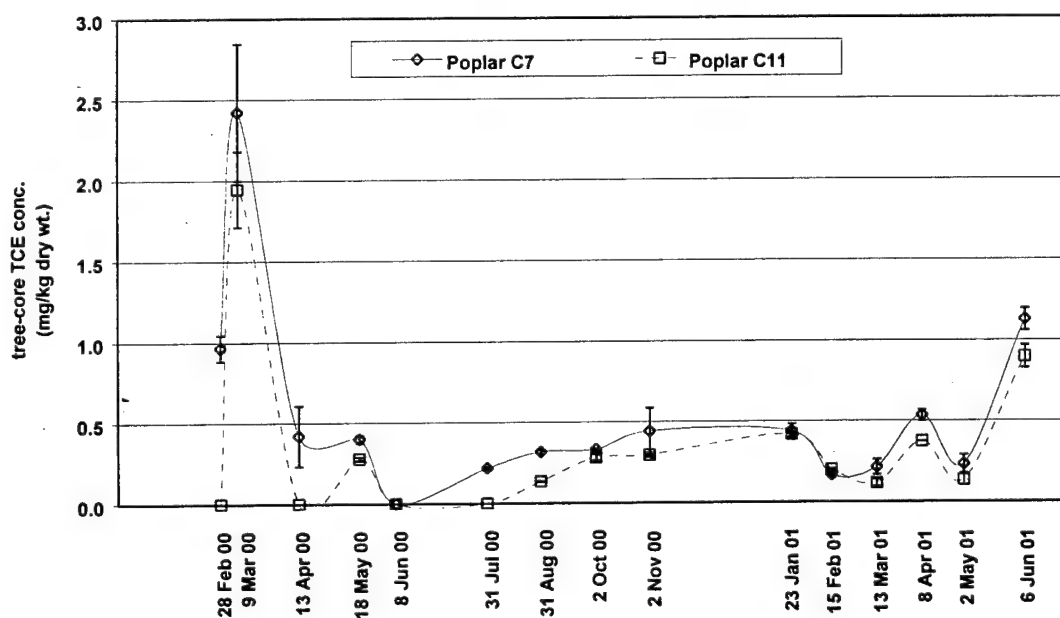
Fig. 4-4.Time series plot of tree-tissue TCE concentrations at site B (medium TCE concentrations in groundwater). Error bars denote $\pm$1 standard deviation.
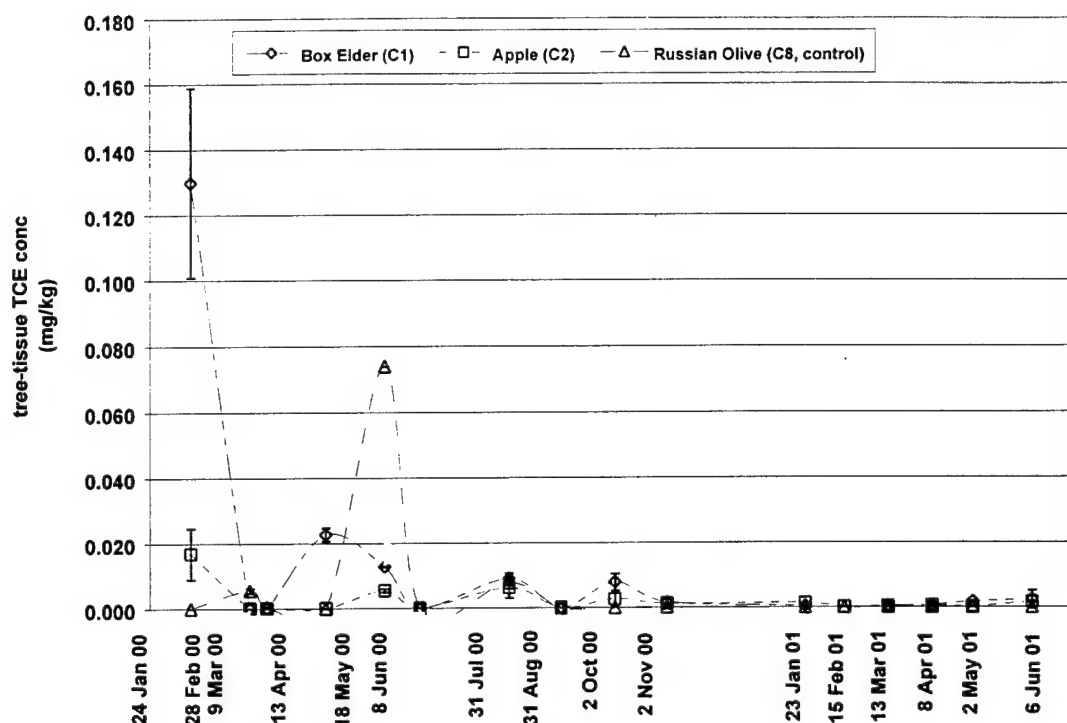
Fig. 4-5.Time series plot of tree-tissue TCE concentrations at site C (low TCE concentrations in groundwater). Error bars denote ±1 standard deviation.

this russian olive, so the result is only speculative.

Nietch et al. (1999) reported that a seasonal TCE concentration change can be explained using seedling water use. Seedling water use is dependent on stomatal opening. Several environmental factors including radiation intensity, relative humidity, and temperature trigger a leaf's guard cells to open and close the stomates (Taiz and Zeiger, 1991). Long, hot, dry days are expected at HAFB during the summer months. Consequently, seasonal tree-tissue TCE concentration trends are expected. To evaluate potential weather effects on tree-core concentrations, daily precipitation and maximum temperature data (Table E) from nearby weather stations (Ogden Pioneer P H and Ogden Sugar Factory) were obtained through the National Climatic Data Center (Asheville, NC) via the internet (NCDC, 2001).

HAFB weather station data were not available at the time of the data collection. Consequently, two Ogden weather stations were used to estimate tree-tissue concentration trends possibly affected by temperature or precipitation (data listed in Appendix E). Other parameters (e.g., cloud cover, solar radiation, humidity) would have been useful but were unavailable at the time of the data request. Daily maximum temperatures did appear to follow the column contours in Fig. 4-6. Precipitation did not appear to have any effect on tree-tissue concentrations.

A review of well data provided by Hill AFB Environmental Management (Table C) revealed relatively constant depths to groundwater. The only dramatic change in groundwater level was in well U4-006 (near the cottonwood showing dramatic increases in June). Depth to groundwater dropped 2 ft in one month and may be indicative of
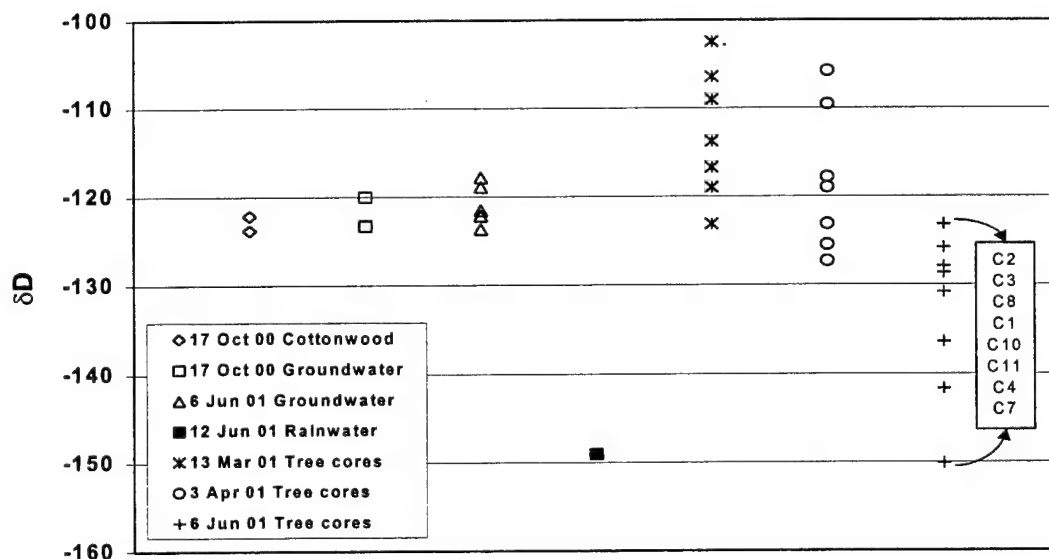
Fig. 4-6. Time series plot of large cottonwood at site A with an overlay of daily maximum temperature and precipitation data.

higher transpiration rates and/or surface flux. However, more data are necessary to make any conclusions regarding groundwater elevation and its effect on tree-tissue concentrations.

Radial Location and Height Effects on
Tree-Tissue Concentrations

Species differences do appear likely as seen in Fig. 4-4 and 4-5. However, these apparent species trends may also be an artifact of tree size as measured by trunk circumference. In most cases, the highest tree-tissue concentrations at the high groundwater TCE concentration site (near wells U4-006 and U4-065, groundwater TCE concentrations > 3700 µg/L) were found (in decreasing order) at the cottonwood, then the russian olive, followed by the poplar. However, the same order is noted for their respective trunk circumferences. Lastly, having earlier shown (Fig. 3-6) a 2X variability for a medium sized poplar dependent on sample location around a trunk, one cannot discredit that apparent species differences may be caused by the sampling method rather than environment. Vroblesky et al. (1999) reported species differences that overshadow radial variability. To define which effect is greater would have required many additional tree cores from each tree. The objectives of this project did not permit monthly sampling in triplicate at multiple sites around a trunk – the risk of irreparable damage (e.g., disease) from nine cores per month was too great.

Finally, Vroblesky et al. (1999) reported decreases in TCE concentration with height in a very large bald cypress tree. Fig. 3-7 contains information on tree-tissue concentrations at different heights for a HAFB cottonwood at Site A that exhibit an

opposing trend – a slight increase with height. The same cottonwood tree sampled two months later exhibited a decreasing concentration up to approximately 5 meters above ground level then an erratic increase at the highest sampling point (Fig. 3-7). Similarly, Pajak et al. (2000) did not note a definitive tree-tissue TCE concentration trend. Because of trunk structure, the last two heights were collected from different sides of the trunk adding another potential source of variance for the height data. Again, conclusions regarding tree-tissue TCE concentration trends dependent on sampling height are contingent on the true source of variability—radial, vertical, or seasonal. More data are necessary to develop a viable hypothesis regarding tree-tissue concentration trends.

Source Water Study Using Stable Isotopes of Hydrogen

Tree cores, groundwater, and rainwater (a single event) were collected to determine the deuterium/hydrogen ratio of tree-core water (xylem). Groundwater δDs remain relatively constant throughout the year (C. Cook, SIRFER laboratory supervisor, University of Utah, personal communication, 9 July 2001). As expected, groundwater δDs remained relatively unchanged. The results in Fig. 4-7 (data in Appendix D) generally show that the tree prefers to use the groundwater source. Several rains occurred between February and June 2001, with the greatest rainfall occurring in mid-April (see Fig. 4-6). As a result of the increased source of surface water, the June 2001 tree cores appear to have begun a δD shift toward the signature of the rainwater (more negative). The individual data points for the June 2001 tree-core stable isotope data do not follow any particular trend with respect to tree species, size, or location. Table 4-1

provides a cross reference for species, location, and size identification using the tree

identifier (e.g., C7 or C10). Results show that stable isotopes of hydrogen may be a

useful tool at HAFB for the determination of a tree's water source. However, only a

general conclusion can be made because a δD range for rainwater should not be defined

with only one rainwater sample.

In conclusion, 18 months of tree-tissue TCE concentration data and 6 months of

groundwater data show promising potential for the development of water sampling

techniques that utilize trees rather than costly, technically advanced equipment.

Additional tree-tissue concentrations taken from trees growing above a wider range of

known groundwater concentrations would fill the gaps shown in Fig. 4-2 and aide in the
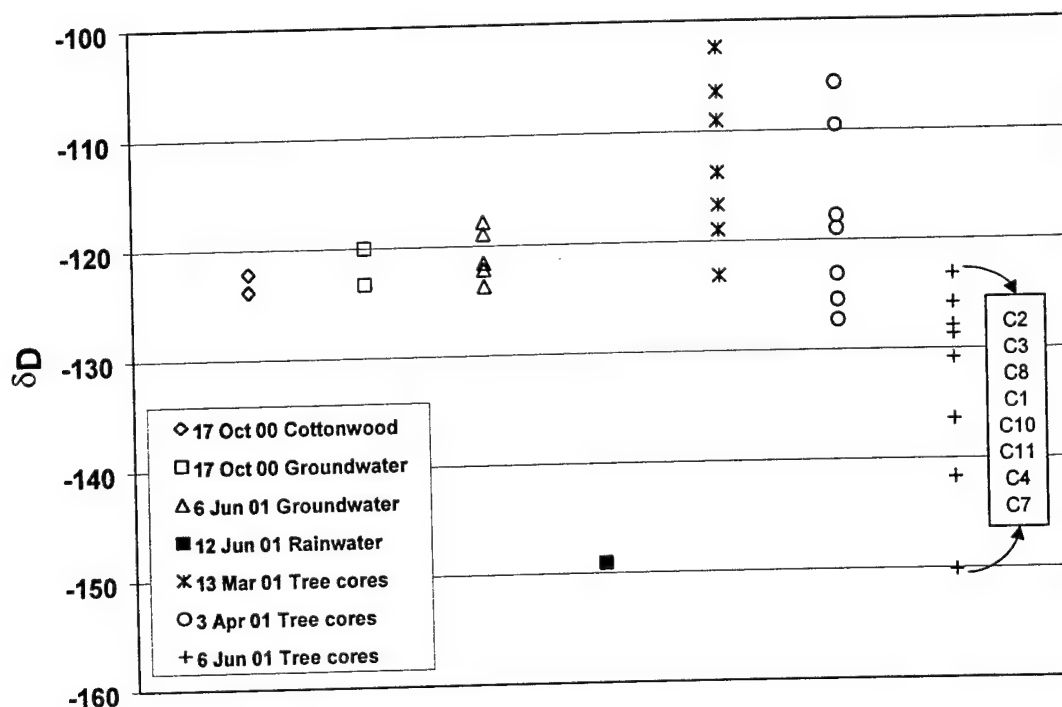
Fig. 4-7. Stable isotopes of hydrogen taken from groundwater, tree-core water, and rainwater. Box to right of June '01 tree-core data contains tree labels for each point in the June '01 tree-core set (top to bottom).

development of a useful predictable relationship. Additional research is necessary to fully evaluate species, seasonal, and radial sources of variation. However, these sources of variance might be overcome by sampling during the same season (ideally the same day), at the same height, fully around the circumference (to obtain an average trunk concentration), and from trees of similar size.

Based on personal field and laboratory experiences during the past 18 months, approximately $300 worth of coring and cleaning equipment, two people, and a days worth of work can result in the collection of well over 100 tree-core samples. Within 48 h of collection, these samples can be analyzed using automated HSA equipment without having to take any significant sample preparation steps (as are required in the purge and trap techniques). At the very minimum, the general relationship as shown in Fig. 4-2 could be used at HAFB OU-4 as a tool for site selection of additional groundwater wells (so long as the tree is of sufficient size to be tapped into the groundwater). If a groundwater/tree-tissue concentration relationship were fully developed and tested, the use of tree-tissue sampling (and subsequent HSA) could delineate the extent and magnitude of a shallow groundwater plume for less money (e.g., less drill rigs and less roads) and with less training.

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CHAPTER 5

A SORPTION/DESORPTION STUDY OF COTTONWOOD AND RUSSIAN OLIVE TISSUES FROM HILL AFB SITE OU-4

Abstract

The sorption and desorption of trichloroethylene (TCE) to wood tissue was characterized. Two trees, both heavily monitored for plant uptake of groundwater contaminated with TCE, were selected in an effort to obtain site-specific information on the partitioning of TCE between tree tissue and water. A sealed batch technique utilizing $[^{14}C]$ TCE, headspace vials, and liquid scintillation counting was shown to be effective for short term mass balance determination of wood-water partitioning coefficients, K_{Wood}. However, the measurement of only the aqueous phase during the sequential dilutions (making estimates regarding volatilization losses) resulted in substantial overpredictions of tissue $[^{14}C]$ TCE concentrations and were not effective for the determination of single point K_{wood} values. Sorptive equilibrium was achieved within 24 h for water saturated wood shavings collected using a ½ inch wood drill bit from cottonwood (*Populus deltoides*) and russian olive (*Elaeagnaceae elaeagnus*). Sorption to both tree species exhibited linear sorption isotherms. Desorption coefficients were determined by mass balance measurements of both the aqueous and solid phases at the start and end of the sequential dilutions. Desorption isotherms were shown to be linear and reversible. Wood-water partition coefficients were 16.4 mL/g for cottonwood and 11.51 mL/g

for russian olive. **Normalized to lipid content the cottonwood measured log K_{lipid} of 3.36 and the russian olive measured log K_{lipid} of 3.45.**

Introduction

The Hill Air Force Base (HAFB) Environmental Management Directorate, responsible for the cleanup of all groundwater contamination on the base property, has been continually evaluating new and innovative technologies for the removal of TCE. Three projects that include a laboratory plant uptake study, and two field plant uptake studies (HAFB and Cape Canaveral Air Station, Florida) sparked an increased interest in the evaluation of the use of trees (commonly called phytoremediation) as a viable treatment alternative at HAFB OU-4 (Doucette et al., 2000; Hayhurst, 1998; Orchard, 1998; Pajak et al., 2000). This ongoing research is evaluating all potential TCE removal mechanisms including but not limited to: phytovolatilization, metabolic degradation, rhizospheric degradation, and soil surface flux. With TCE translocation highly dependent on the specific interaction characteristics between tree tissue and TCE, a sealed batch method was used to determine tissue-water partition coefficients (K_{Wood}) for both sorption and desorption using [^{14}C]TCE. Two specific tree-tissue types at site OU-4 were evaluated: cottonwood (*Populus deltoides*) and russian olive (*Elaeagnaceae elaeagnus*).

Trichloroethylene (TCE) is a groundwater contaminant that, as of 1997, has been found in over 60% of the 1430 National Priority List hazardous waste sites (USPHS, 1997). A report posted in 1989 (compiled from federal and state surveys) further developed the extent of contamination by stating that 9 to 34% of water supplies in the

U.S. are contaminated with TCE (USPHS, 1989). Hill Air Force Base (HAFB), located approximately 25 miles north of Salt Lake City, UT was not immune to the extensive use and poor disposal practices of major aircraft repair facilities. Many solvents and degreasers were disposed in open pit landfills at the northern edge of the base property creating what is now known as clean up site Operable Unit 4 (OU-4).

TCE is the principal contaminant at OU-4 – the only volatile organic chemical (VOC) consistently detected in groundwater and seeps at concentrations that exceed its maximum contaminant level (MCL) of 5 μg/l. Approximately 69 acres have become contaminated by TCE as determined from analytical results of samples collected through July 1999. Most TCE contamination has been detected in the upper 30 ft of the shallow aquifer (ideal for potential use by deep-rooted trees in the semiarid environment near the Great Salt Lake) with depths to groundwater varying from 5 to 35 ft.

Plant structure influences the uptake and transport of xenobiotics like TCE from pore water in the rhizosphere into the root structure. Compounds entering plant roots can move in the inter- and extracellular spaces of the cortex until reaching the endodermis. The endodermis, a cylindrical sheath of cells tightly bound together by the lignified Casparian strip, surrounds the vascular tissues of the plant. TCE must pass through this regulatory barrier before having access to the vascular tissues where significant translocation can occur (Bromilow and Chamberlain, 1995). The octanol-water partition coefficient (log K_{ow}) and transpiration stream concentration factor are two primary terms that have been used to describe plant-contaminant interactions.

The term transpiration stream concentration factor (TSCF), first defined in 1974 by Shone and Wood (1974), was a tool created for use in the comparison of chemical

partitioning within plant species. The TSCF is a measure of the efficiency of this movement of a contaminant from the roots to the shoots via the transpiration stream – a ratio of the concentration in the transpiration stream to the concentration of the chemical in the external soil solution. As TSCFs approach unity, the compound is said to move with the same efficiency of water (unless significant degradation is occurring within the plant).

Briggs et al. (1982) reported the first predictive relationships for the plant uptake of contaminants using the logarithm of the compound's octanol-water partition coefficient, or log K_{ow}. Significant work on the relationship between lipophilicity (estimated using log K_{ow}) and TSCFs has shown that compounds having log K_{ow} values around 1.8 [for barley shoots; (Briggs et al., 1982)] and 3.07 (for de-topped soybean plants; (Hsu et al., 1991) had optimum TSCF values. Although Bromilow and Chamberlain (1995) did not offer an explanation for these optimum log K_{ow} differences, Orchard (1998) has suggested that the differences between these two optimum log K_{ow} values may be a result of differences in plant age, plant size, or experimental technique. Briggs et al. (1983) conducted controlled growth greenhouse experiments using barley shoots and radiolabeled compounds. They concluded that constant maximum concentrations in stems were rapidly attained (24-48 h) for chemicals of log K_{ow}<3, but were attained more slowly for more lipophilic chemical. The log K_{ow} for TCE has been reported to be approximately 2.53 (Mackay et al., 1992).

Orchard (1998) stated that "compounds that are either highly polar (log K_{ow} < 0.05) or are very lipophilic (log K_{ow} > 4.5) are not expected to be significantly taken up

by the plant." Consequently, detectable levels of TCE (log K_{ow} 2.53) in the tree cores were expected and ultimately measured at HAFB site OU-4.

Sorption coefficients describe the equilibrium distribution of a chemical between two phases, in this case tree tissue and water (K_{wood}). Sorption coefficients are key input parameters in models used to predict contaminant behavior in the soil subsurface and are used as guide in the decision-making process (Doucette, 2000). Likewise, K_{wood} may provide critical information for use in future decision making regarding the use of trees as a remediation alternative.

Several sorption processes have been reported. They include van der Waals and dipole-dipole interactions, H-bonding, ionic interactions between charged species, and site-specific chemical bonding (possibly irreversible) (Schwarzenbach et al., 1993). Several papers have been published specifically targeting sorptive effects to the basic polymeric components of wood: lignin (25-31% of softwood mass), cellulose (40-44% of softwood mass), and hemi-cellulose (remaining mass) (Thompson, 1996). Mackay and Gschwend (2000) have shown that K_{wood} can be estimated with values of the wood fractional lignin content and the reported $K_{lignin} - K_{ow}$ free energy relationship. Furthermore, Trapp et al. (2001) have shown that K_{wood} is highly correlated to log K_{ow} and concluded that the retention mechanism (due to K_{wood}) may be an effective elimination process for groundwater contaminants.

Partition coefficients for solid/liquid phases of both nonvolatile and semivolatile compounds are determined by adding a known mass of compound into a system, allowing the system to equilibrate, then measuring the aqueous phase concentration (ASTM, 1987). That fraction of the compound not directly measured within the aqueous

phase is assumed to be sorbed to the solid phase. However, this may not be so effective when the compound of interest is a volatile. Losses like volatilization can result in an overestimate of fractions sorbed to the solid phase. By using a modified sealed batch method developed by Benson (2001), this project sought to overcome the volatilization losses by using sealed headspace (HS) vials. An initial kinetic experiment was completed to determine how much time was required for TCE within the solid and aqueous phases to reach equilibrium. By maintaining the samples at a constant temperature ($20 \pm 1°C$ for this experiment) and spiking the sealed system with a set of five different contaminant concentrations, a sorption isotherm was developed whose slope defined K_{wood}.

Materials and Methods

Tissue Preparation

With HAFB permission, large limbs (diameter > 30 cm) were taken from a cottonwood and a russian olive at site OU-4 to the Utah Water Research Laboratory (UWRL) for processing. Using a ½-inch wood bit, several hundred grams of fresh tree stem tissue were collected at cross sections randomly around and along each limb. Not only did the bit speed up the collection process (the other option was an incremental borer and hours of work), but it also created homogenous shavings. Care was taken to prevent overheating of the bit and charring of the tissue. Following collection the tissue was placed in aluminum trays and dried in a 103° C oven for several days until completely dry, then stored in air tight wide mouth glass jars at 4° C until needed. Contamination from TCE present in the tissue at the collection time was not considered a conflict to this project because of the volatility of TCE and the planned use of [^{14}C] TCE.

Experiment Preparation

The basic plan was to place approximately 1 g of tissue in a sealed headspace vial with a known volume of deionized water and spike the system with a known mass of [^{14}C] TCE (the only exceptions were for blanks). The radiolabeled TCE was of certified purity (> 98%) obtained from Sigma Chemical Co. (St. Louis, MO). The experimental design involved the use of three sampling sets (water only, russian olive, and cottonwood). Each set contained triplicate samples spiked at five separate concentrations of [^{14}C] TCE (approximately 11 mg/L, 5.5 mg/L, 0.98 mg/L, 0.44 mg/L, and 0.10 mg/L). Each set also contained a triplicate set of blanks and a triplicate set of [^{14}C] pyrene-spiked samples at approximately 0.025 mg/L for quality control. The headspace vials (22 mL, Kimble Glass Inc., NJ) were sealed with aluminum crimp-on caps lined with silicon rubber/PTFE-lined septa.

Sorption Kinetics

Three sets of headspace vials containing cottonwood tissue, russian olive tissue, and water, respectively, were spiked to approximately 1 mg/L with [^{14}C] TCE and placed on a rotary tumbler (~30 rpm) in a constant temperature room (20 ± 1°C). Small aliquots (5 or 10 μL) of each aqueous sample were collected through the septa with a 25-μL gas-tight syringe at 3, 6.75, 12.75, 24.25, 48.5, and 72.5 h. Each aliquot was added to a 7-mL scintillation vial (Fisher Scientific, Pittsburgh PA) containing 7 mL of Beckman Ready Gel ® (Beckman Instruments, Inc., Fullerton, CA) then counted by liquid scintillation counting (LSC) using a Beckman LS6000SE Liquid Scintillation Counter. Average concentrations from each set of replicates were normalized to initial concentration and plotted against time. An equilibration time was selected when the change between times

was less than 5% in the solution concentration.

Sorption Procedure

Those headspace vials containing tissue were spiked with an appropriate amount of [^{14}C] TCE to reach the targeted concentration (~0.1, 0.44, 0.98, 5.5, and 11 mg/L), then placed on the rotary tumbler for 24 h as determined by the kinetics experiment above. After removal from the tumber, each HS vial was centrifuged to settle the finer particulate. A 2-mL sample was collected from each HS vial using a 2.5-mL gas-tight syringe (Hewlett Packard, San Fernando, CA), placed into a 7-mL scintillation vial containing 5 mL of Beckman Ready Gel $^®$, then counted by LSC. The remaining aqueous solution in each sorption HS vial was decanted off and discarded. Duplicate and/or triplicate subsamples were collected and counted approximately every tenth tube.

Methanol Extraction and Combustion

Methanol extraction was used to remove any remaining extractable [^{14}C] TCE after decanting of the aqueous solution in the previous step. Each HS vial was filled with ACS grade methanol (Fisher Scientific, Pittsburgh, PA) minimizing headspace and recapped with a new aluminum crimp-on cap. Following a 24-h equilibration time on the rotary tumbler (again in the constant temperature room), the HS vials were centrifuged, sampled (2 mL), and counted by LSC as in the sorption procedure. Following methanol extraction all of the vials were left open to dry under the ventilation hood. Once completely dry, the tissue samples were each split into approximate thirds by mass (~ 0.33 g) and combusted using an R.J. Harvey Oxidizer (R.J. Harvey Instrument, Hillsdale, NJ). During combustion the [^{14}C] TCE is evolved into [^{14}C] CO_2 and trapped

in a 20-mL solution consisting of 50% Ready Gel®, 40% methanol (ACS grade), and 10% monoethanolamine (ACS grade, Fisher Chemical Phillipsburg, NJ). Resultant trapping solution was poured into 20-mL scintillation vials (Fisher Scientific, Santa Clara, CA) and also analyzed by LSC.

Desorption Procedure

The desorption procedure was designed such that its first 24 h were identical to the sorption procedure. However, upon collection of the 2-mL subsample for analysis of aqueous phase concentrations an additional 3 mL of sample was collected and discarded (except for use as an occasional duplicate). Following this overall removal of approximately 5 mL of aqueous solution (~ 25% of total solution volume), clean deionized water was used to replace that which was removed. This resulted in an approximate dilution ration of 75% at each sequential dilution step. Five dilution steps were accomplished (one every 24 h). Finally, on what would be the sixth dilution after 144 h of desorption, the aqueous solution was sampled (2 mL) and the remainder discarded. Methanol extraction and tissue combustion followed thereafter as discussed in previous steps.

In order to keep the vials sealed to minimize volatilization losses during the dilution steps, a second smaller needle was used to allow the displacement of headspace during the removal and replacement of the aqueous solution using the other needle. Two needles were used so that while one was removing the 5 mL of solution the other needle was preventing the formation of a vacuum in the HS vial and vice versa. Special care was taken to minimize punctures in the septa by reusing the same holes as much as possible.

Lipid Extraction

A modification of EPA Method 3540C, Soxhlet Extraction, and Method 3500B, Organic Extraction and Sample Preparation, previously used for determination of lipids in worms Benson (2001) was used to extract lipids from several trees at site OU-4. All glassware was precleaned with methanol, oven dried overnight at 103°C, then tarred prior to use (gloves were worn during all procedures). Tree tissue was placed in a 70°C oven and slowly dried. Weights were monitored until core weights did not change by ±0.02 g. The tree cores were ground using a coffee grinder in preparation for the soxhlet extractions. Ground tree tissue was placed in each extraction thimble along with approximately 8 g of Na_2SO_4 to absorb any remaining moisture in the system. Several quality control samples were also prepared. One duplicate tree-tissue sample was used for a matrix spike using approximately 0.1 g of vegetable oil. Vegetable oil was used in a thimble without tree tissue for determination of simple mass recovery. Finally, a reagent blank using a thimble and approximately 0.1 g of the Na_2SO_4 was used to identify any laboratory contamination. Approximately 100 mL of ethyl ether was used in the soxhlet extraction apparatus for approximately 20 h of operation. Rheostats were set to level 2 (approximately 60°C) to obtain 1 cycle per 10 min within the closed system apparatus. All work was completed under an appropriate laboratory hood. Detailed gravimetric records were kept for all mass added or ultimately removed from the system.

Results and Discussion

Before this sorption experiment could begin, a preliminary test was necessary to determine how much time is required for the sealed system to reach equilibrium. Using a

single midrange concentration (~ 1 mg/L [^{14}C] TCE), three separate systems (russian olive, cottonwood, and a blank control) were monitored over a 72-h period using LSC to determine when the HS vial systems had reached equilibrium. Fig. 5-1 shows the resulting curves (normalized to initial concentration). Supporting data are listed in Table G.1 of the appendix. Because the change in concentration was less than 5% from the 12- to the 24-h sampling events, a 24-h equilibration time was chosen for the course of this sorption/desorption project.

Both 24-h wood sorption isotherms (Fig. 5-2) appear to be linear. However, a single cluster of data points appears below the linear regression line. For comparison, a Freundlich model was used which resulted in a slightly better fit to the data (Fig. 5-3 and
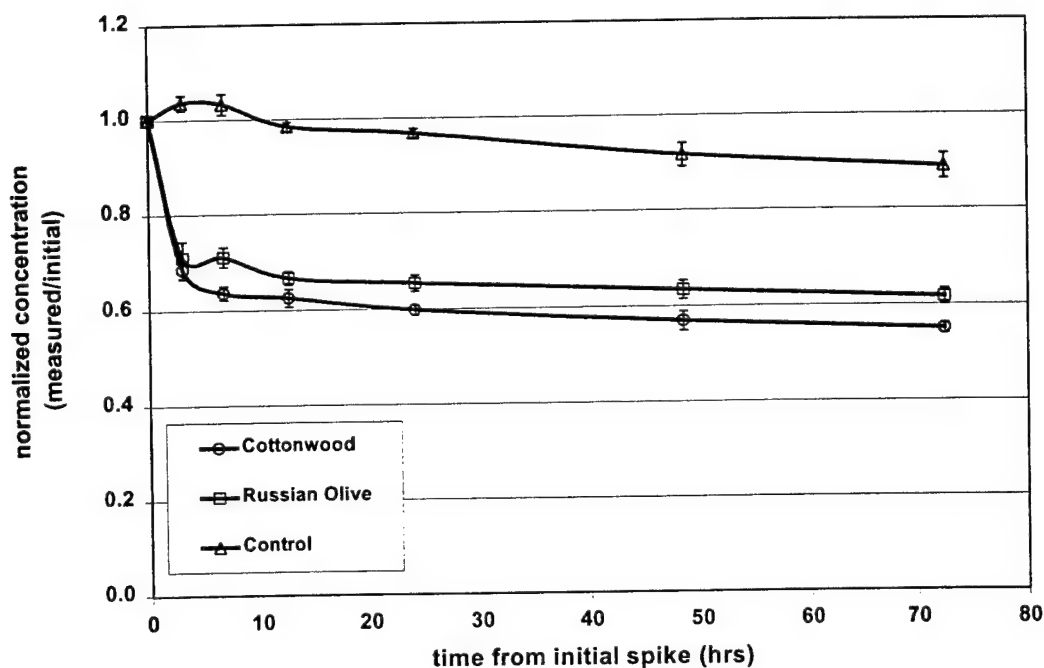
Fig. 5-1. Sorption kinetics test data summary for cottonwood and russian olive tissues from OU-4 (± one standard deviation). All data normalized to initial concentration. Kinetic data can be found in Table G.1.
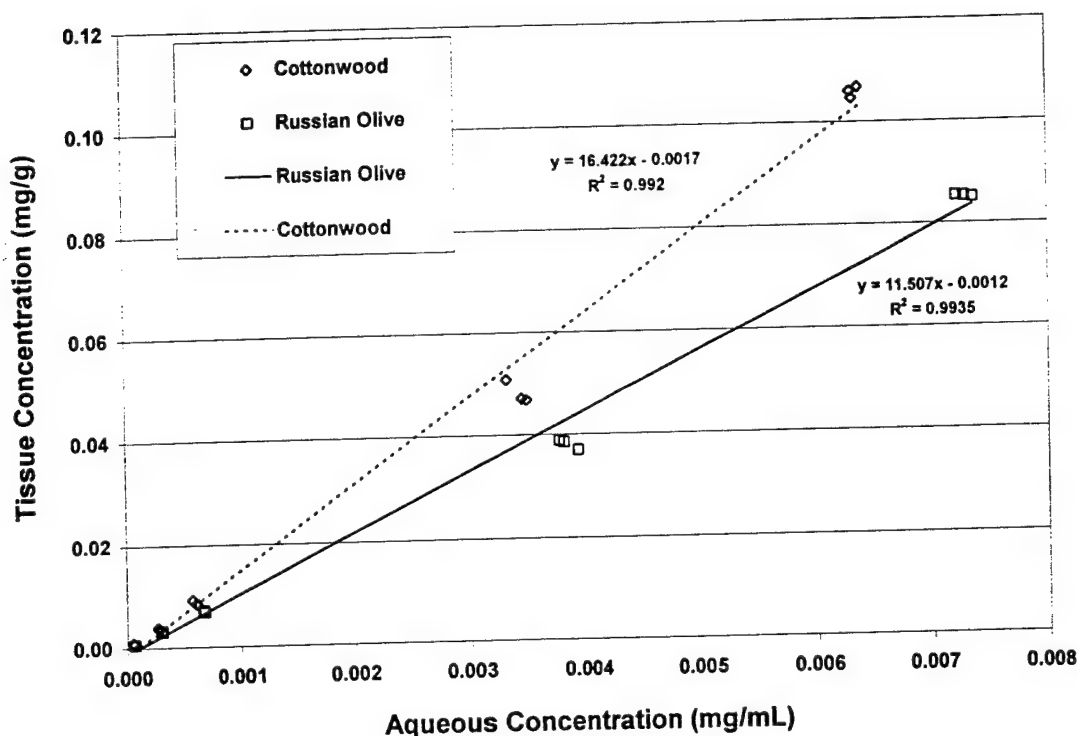
Fig. 5-2. Comparison of TCE sorption isotherms using linear regression. Cottonwood and russian olive tree tissue taken from Hill AFB OU4.

5-4) as determined by an evaluation of residual plots. Values of n and K_f from the cottonwood and russian olive Freundlich isotherms are listed in Table 5-1. These n-values were obtained from the slopes of the lines in Fig. 5-3 and 5-4. For any Freundlich isotherm, the isotherm is convex for n < 1, linear for n = 1, and concave for n > 1. As seen shown by the 95% confidence intervals shown in Table 5-1, the cottonwood Freundlich isotherm was not significantly different from a linear regression model (where n = 1). However, the russian olive Freundlich isotherm 95% confidence interval did not include the number one. The russian olive Freundlich isotherm was statistically different from a linear regression with a range of n from 1.017 to 1.057. Although different from one, the range of n is different by only a small margin. The russian olive data are better
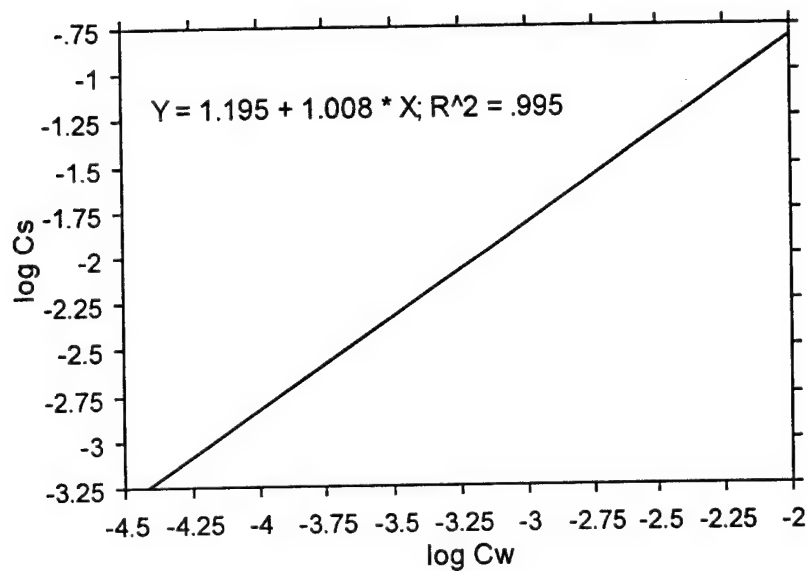
Fig. 5-3. Cottonwood Freundlich isotherm (20 ± 1°C) with 95% confidence intervals (n = 15).
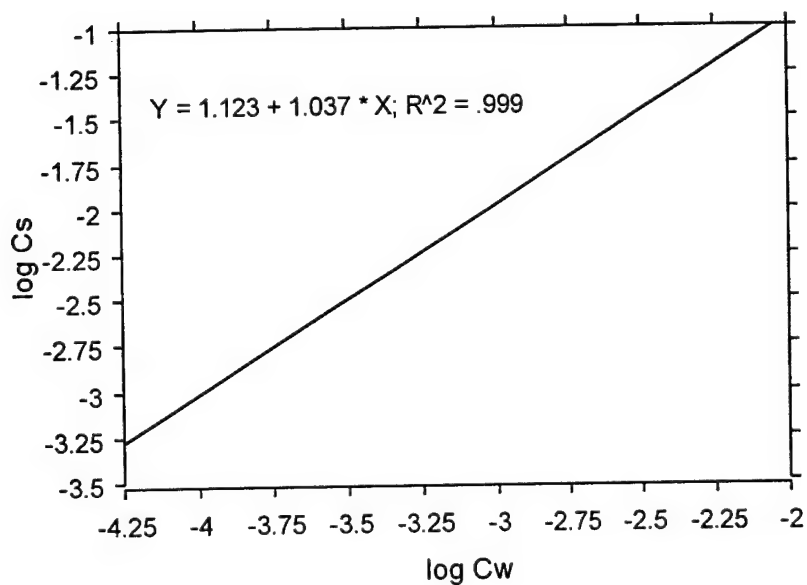
Fig. 5-4. Russian olive Freundlich isotherm (20 ± 1°C) with 95% confidence intervals (n = 15).

fit by a Freundlich isotherm; however, the linear regression forms will be used for simple estimation of the K_{wood} values. Use of a linear regression is supported by other research literature. Mackay and Gschwend (2000) reported similar results stating that the 95% confidence limits for their n values included one and were therefore not significantly different from the linear model. All sorption data are listed in Table G.2.

With a K_{wood} consistently higher than the russian olive, this sorption coefficient alone may explain why the cottonwood consistently returns higher concentrations of tree-core TCE. Percent recovery for $[^{14}C]$ TCE during the sorption experiment was 96.4 ± 4.3% for n = 30. Lipid extraction results returned lipid fractions of 0.00721 and 0.00413 for the cottonwood and the russian olive, respectively (Table G.3). Dividing the derived K_{wood} values by the known fraction of lipid, a K_{lipid} can be derived. Values for K_{wood} taken from the slopes of the isotherms within Fig. 5-2 and 5-3 were 16.42 mL/g (cottonwood) and 11.507 mL/g (russian olive). Consequently, their K_{lipid} (calculated) values were 2277.7 mL/g and 2786.2 mL/g. In terms of log K_{lipid} (a more comparable value with current literature), the cottonwood had a log K_{lipid} of 3.36 while the russian olive had a log K_{lipid} of 3.45. Table 5-2 provides data for comparison with current literature values. In Table 5-2, the single point K_{wood} values are defined as the values of

Table 5-1. Freundlich isotherm summary table.

Tissue type	n	95% confidence interval	K_f	95% confidence interval
Cottonwood	1.008	0.966 – 1.049	1.123	1.060 – 1.186
Russian olive	1.037	1.017 – 1.057	1.195	1.060 – 1.330

Freundlich Equation: $C_s = K_f C_w^n$, where Cs (TCE concentration in solids, mg/g) and Cw (TCE concentration in water, mg/mL)

the individual points within all linear isotherms shown in this chapter.

Both Mackay and Gschwend (2000) and Trapp et al. (2001) have shown strong relationships between K_{wood} and log K_{ow} and have advanced the modeling of contaminant transport (or lack of) within the structure of a tree. Using K_{wood}, molecular diffusivity, and an internal wood solid-to-water ratio, Mackay and Gschwend (2000) proposed a working model capable of order of magnitude estimates of wood diffusion coefficients.

Fig. 5-5 and 5-6 show convincing evidence that desorption is reversible following the same pathway as the sorption data. Initially, all data points collected between the 24-h desorption and 144-h desorption step were included in the sorption isotherm. However, accounting for evaporative (and other) losses was leading to the wrong conclusions. Assuming no evaporative losses, a total TCE mass balance can be made at

Table 5-2. Summary of single point K_{wood} values from sorption/desorption study.

| | | Single-point K_{wood} values (mL/g) | |
		Sorption	Desorption
OU-4 Cottonwood TCE	Mean	12.94	11.80
	SD	1.23	2.41
	Range	9.77 – 15.33	8.36 – 14.72
OU-4 Russian olive TCE	Mean	10.28	8.13
	SD	0.46	3.08
	Range	9.39 – 10.95	5.17 – 14.07
Calculated using † TCE log K_{ow} (Mackay and Gschwend, 2000)	Range	6.7 - 61	na

† log K_{lignin} = (0.74 ± 0.09)log K_{ow} – (0.04 ± 0.25), K_{wood} = $f_{lignin}K_{lignin}$, assuming f_{lignin} = 0.3 for softwoods
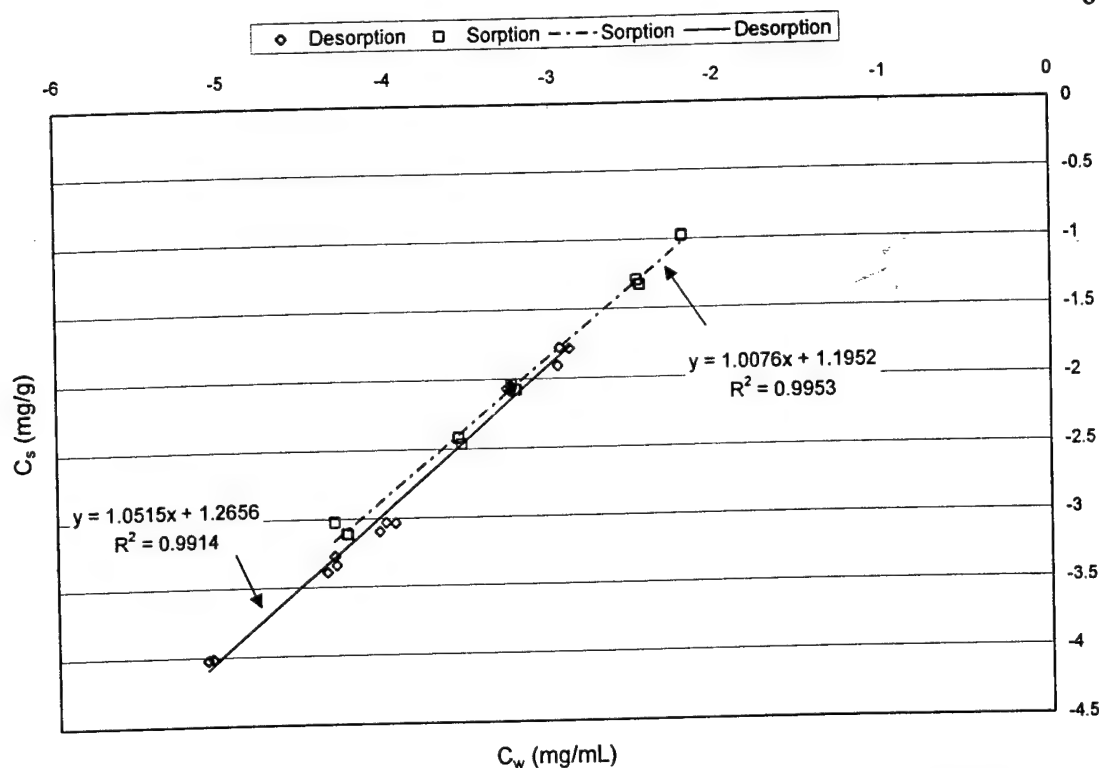
Fig. 5-5. Sorption and desorption Freundlich isotherms for the cottonwood. C_s (tissue TCE concentration), C_w (water TCE concentration).

each successive step because the exact amounts of aqueous solution removed and added are known. If no other losses are assumed, then any mass not detected in the aqueous solution must be in the solid tissues. Literature has supported the expectation of a linear and reversible desorption (Mackay and Gschwend, 2000). Poor [14C] TCE recoveries were not expected due to the careful steps taken in the desorption process. However, in retrospect using multiple syringes to more easily transfer sample out and dilution water in may have created too many punctures in the septa through which the TCE could escape. Based on his more successful [14C] TCE recovery, more careful attention to the details of the methods used by Benson (2001) may result in an improved tissue sorption data set.

In summary, contaminant movement is a critical part of the overall soil/plant/air

continuum and a necessary part of any in-depth evaluation of all potentially beneficial effects of phytoremediation. Site specific sorption coefficients were fairly easy to obtain. However, improvements in the dilution and collection techniques are necessary if more trustworthy data is desired between the initial concentration and the final dilution. A larger experiment might use additional replicates during the middle of the desorption period so that some samples might be sacrificed early for quantifiable, mass balanced measurement.

The magnitude of K_{wood} can have dramatic effects on the movement of TCE. During high transpiration seasons, a higher K_{wood} will produce more retardation of TCE
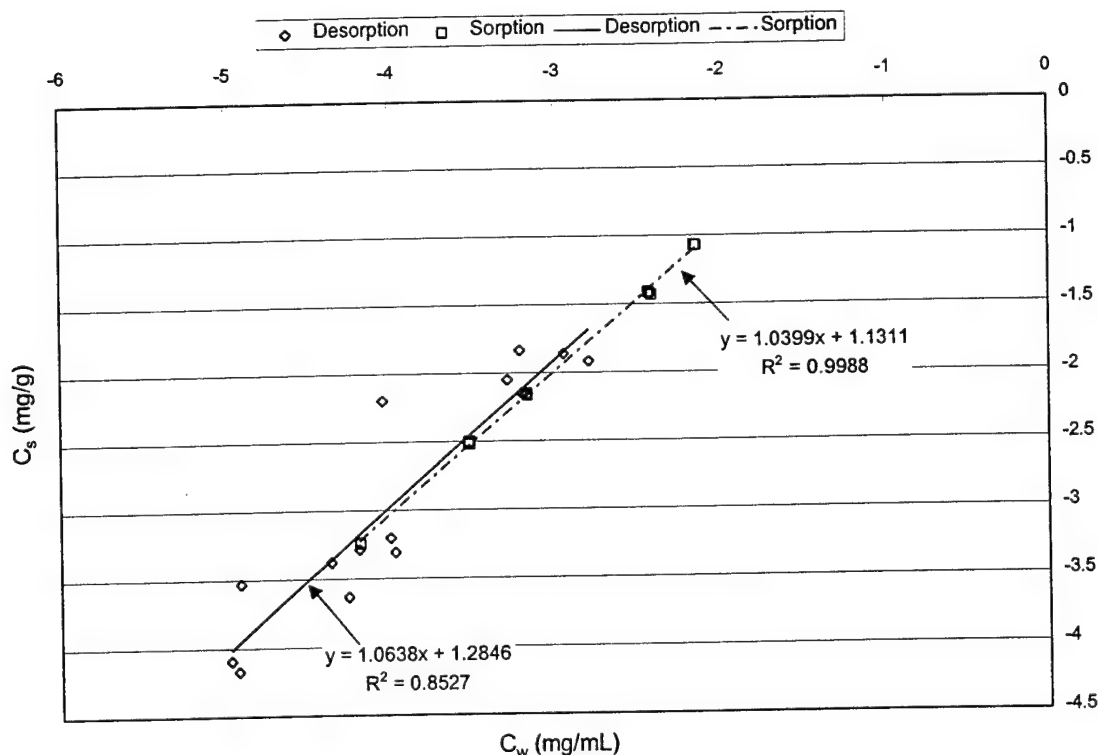
Fig. 5-6. Sorption and desorption Freundlich isotherms for the russian olive. C_s (tissue TCE concentration), C_w (water TCE concentration).

movement possibly causing an increase in tree-tissue TCE concentrations. Furthermore, K_{wood}, by definition, creates immediate effects on other important measurements including the transpiration stream concentration factor (or TSCF). As K_{wood} increases, more TCE moves from solution and becomes sorbed lowering the measurable aqueous concentration, consequently the TSCF drops.

Finally, this study of sorption and desorption coefficients provides explanations for phenomena that have been observed during the past 18 months of tree-tissue TCE sampling and analysis. First, sorption explains why spike recoveries early in the project were so poor. Second, sorption (and especially desorption) explain how transpiration rates might result in changes in the tree-core concentration of TCE.

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CHAPTER 6

CONCLUSIONS

The focus of this project was to evaluate the feasibility of using trees as a cost-effective groundwater sampling and plume delineation tool. This involved the collection of plant tissue and groundwater samples for the analysis of TCE. Headspace analysis using gas chromatography was benchmarked against a more rigorous extraction and analysis technique (methanol extraction, purge and trap/gas chromatography) for performance comparison. Several sample collection and preservation techniques were compared and evaluated (cores vs. shavings, pH 2 vs. pH 10 modifier solutions). Resulting data were used to evaluate sources of tree-tissue TCE concentration variance (radial and vertical sampling locations, seasonal effects, and species effects). To verify the studied trees' use of groundwater (as opposed to other source waters), stable isotopes of hydrogen from groundwater, rainwater, and tree-core water were analyzed and compared. Toward the end of the project, a desire to better understand TCE movement (and potential storage) within tree tissues led to a site-specific evaluation of sorption and desorption coefficients for two species of trees at OU-4 using a sealed batch method and [^{14}C] TCE. These investigations into the relationship between groundwater and tree-tissue TCE concentrations provided supporting documentation for the feasibility of using trees as a cost-effective groundwater sampling tool.

The results of this project have led to a number of findings, which are summarized below:

1. Although tree-tissue TCE concentration variability exists within any single tree, the total variations were not greater than one order of magnitude. Furthermore, a useful

relationship between groundwater and tree-tissue TCE concentrations was shown in Fig. 4-2. With groundwater TCE concentrations shown to be very consistent, the tree variability does not impede on the beneficial use of tree coring as a useful tool for groundwater plume delineation. At the very least, the use of tree coring and headspace analysis can be used for better site selection for well placement.

2. Headspace analysis (GC/ECD) using pH 10 matrix modifying solution (sodium chloride saturated water) was no different from pH 2 matrix modifying solution.

3. The collection of tree tissue using a drill is not recommended. The increased surface area of the shavings and the increased temperatures during collection (heat from the bit) promote an accelerated loss of TCE before sample preservation in the headspace vials.

4. For tree-tissue samples collected at sites where groundwater TCE concentrations were greater than 800 µg/L, purge and trap analyses detected 2.7 times more TCE in each sample than did the headspace analyses. Assuming that methanol extraction removes as much TCE from the core as is possible, this multiplication factor could be used to predict the actual concentrations (and mass) of TCE in the tree tissue.

5. Statistically significant TCE concentration differences were found for those tree cores collected on different sides of a particular tree. For tight variance tolerances, tree cores should be collected within 3 to 5 cm of each other. For a representative measurement of a tree trunk's TCE concentration, samples should be collected on at least 120° radials around the trunk. Based on actual results from a poplar, samples collected on different sides of a tree could vary by a factor of two.

6. TCE concentrations varied by as much as a factor of six when collected at different heights spanning a vertical distance of over 6 meters. However, if tree-core sampling were used for quick groundwater plume delineation, vertical variation would not be as important as radial variation because samples would most likely be collected near the ground level.

7. With respect to vertical variations, TCE concentrations did not follow a consistent trend. Some results show a general decreasing TCE concentration trend with height. Other results show a general increase in TCE concentration with height.

8. Tree-core TCE concentrations tend to slightly increase during the day and decrease at night. However, more data are necessary to confirm the results.

9. Tree-core TCE concentrations varied month to month and may be related to seasonal conditions (which consequently would effect transpiration rates). Data from June of 2000 and 2001 were up to nine times greater than measurements taken the preceding month. However, more data are necessary to determine if this variation is being caused by seasonal trends or by radial location of sample collection on the trunk.

10. Stable isotopes of hydrogen show tree-core water δD signatures that are within the same range of the groundwater signatures. After a period of increased rain, the δD of the groundwater showed a slight decrease in δD approaching the rainwater signature. Differences between the δD of rainwater and groundwater were large enough to distinguish. Therefore, future use of stable isotopes for source water delineation at Hill AFB site OU-4 is recommended.

11. Wood-water partition coefficients (K_{wood}) were determined for a cottonwood (16.42 mL/g) and a russian olive (11.51 mL/g). Tree-tissue lipid content was determined to

be 0.72% and 0.41%, respectively. Consequently, log K_{lipid} values of 3.36 and 3.45 were calculated.

12. The wood sorption and desorption isotherms were linear. Desorption was shown to be reversible, following along the sorption line.

13. The use of headspace vials for sealed batch sorption experiments worked very well (96.4 ± 4.3 % recovery of $[^{14}C]$ TCE). However, very poor recoveries plagued the desorption experiment and were due to the repeated puncturing of the septa during the sequential dilution steps.

Recommendations

Recommendations for improving upon this project would primarily focus on the further evaluation of the sources of tree-tissue TCE concentration variance. Further sampling could focus on the predominant variance for samples collected at ground level (e.g., does radial location variance dominate seasonal variance, or vice versa?). Site selection could help to narrow the focus. For example, a site containing many tree species of similar size and located near groundwater sampling wells would be useful for evaluating species differences. Also, a different site may offer opportunities for sampling trees of the same species and similar size to evaluate seasonal differences. Further investigations using tree cores could be conducted within the confines of OU-4, or expanded to other operable units at HAFB, or further expanded to sites off base. For real-world application, data obtained through this project and/or future projects combined with available geographical information software like ArcView™ might transform the

data into a more powerful tool for well placement or groundwater contamination related decisions.

Although cooperation and discussion were primarily kept to the confines of Utah State University and HAFB for this project, a team approach that reaches outside the current umbrella might lead to more data for comparison. With only two primary groups [Utah State University and D. Vroblesky's team (Vroblesky et al., 1999a, 1999b)] actively pursuing the use of tree-core sampling as a groundwater sampling tool, a cooperative relationship between the two teams for comparison of data might lead to an expansion of data and ideas.

Engineering Significance

It has been well established through extensive research that contradictory observations abound regarding the uptake and translocation of volatile organic compounds (especially TCE) by plants (Orchard et al., 2000). As a part of the overall evaluation of the soil-plant-air continuum, quantifiable tree-tissue TCE concentration analysis is absolutely necessary for making conclusions regarding the fate of TCE as it moves from the groundwater, through the stem, and reaches the leaves.

The comparison between the headspace analysis method and the methanol extraction/purge and trap analysis method (assumed to be the most rigorous TCE extraction technique for total mass determination) was made to determine how quantifiable headspace analysis really is. Personal experience gained during this "head-to-head" method comparison places headspace analysis way ahead of purge and trap when ease of use, minimal time, and minimal training and experience are the deciding

factors. This project demonstrates that a quick and quantifiable tree-tissue TCE concentration analysis method exists for those who have access to an incremental borer and some basic laboratory equipment. For a real-world determination of the engineering significance of quantifiable data, the follow example is presented.

Proposed by Orchard (1998), this example assumes a site is contaminated with 10 mg/L of TCE. Assumptions include: 1500 trees/acre, 3 acre ft/yr of total transpired water, groundwater as the primary water source, 1000 kg/acre initial TCE concentration, 100 kg/acre TCE clean-up goal, and first order kinetics. Orchard calculated the approximate TCE removal time required to reach the preset goal. Depending on which transpiration stream concentration factor (TSCF) is used, the cleanup time ranged from 89 to 346 years. In the hands of a decision maker or regulator, this difference of 257 years can be the final determining factor for which type of remediation technology is used. Quantifiable data were necessary for the determination of the TSCF which ultimately resulted in a decision involving the allocation of significant resources (both time and money).

As shown above the ultimate proof of engineering significance is often measured in the amount of time and money saved. On a single day during this project, over 80 tree cores were collected from a large sample set of trees growing near a groundwater seep at OU-4. This tree-core sampling required vials, caps, matrix modifying solution, a cap crimping tool, an incremental borer, a cooler, and labor (with minimal training). Groundwater sampling (where no well previously existed) would require a very large capital investment in equipment, a well trained drilling crew, digging permits, the generation a large volumes of waste (e.g., contaminated groundwater and soil), etc.

The evaluation of any newly discovered groundwater contamination site involves educated decisions for initial site selection of wells. For the environmental company hired to design and then carry out an initial site assessment, the opportunity to sample hundreds of trees in a single day using only a handful of technicians with minimal capital investments would save them literally thousands of dollars.

For the determination of the TSCF in the above example, a quantitative analysis of TCE in the tree tissue would be necessary. Time and money savings associated with the use of a tree-tissue headspace analysis method (instead of purge and trap) are more difficult to ascertain. With purge and trap, there are additional costs not associated with headspace analysis. Purge and trap methods require the use of an extraction solvent that is then used to generate a dilute sample within purge and trap grade water. Wastes generated include both the large quantity of methanol solvents and the remaining diluted sample. Based on personal experience, time and the level of technical expertise required for operation are less for headspace analysis but are more difficult to measure. However, one may not be able to analyze the tissue any easier than by simply loading the headspace vial containing an easily obtained tree core onto an auto-sampling rack and pressing start.

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APPENDICES

Appendix A.

Monthly Tree-Core TCE Concentration Data

Table A.1 January 2000 tree-core TCE concentration data.

Sample Type	x (ppb) *	y (area)	wet core wt (g)	dry core wt (g)	% water	mass TCE (ug)	ug/g (TCE in dry core)
			Head Space				
2	2.40	8603					
5	5.04	16773					
10	11.76	36109					
25	27.04	74374					
50	54.18	130400					
100	96.04	200440					
modifier blank #1	ND	0					
c1a	13.52	40871	1.954	0.842	56.91%	0.135	0.16
c1b	9.62	30163	2.172	0.936	56.91%	0.096	0.10
calibration check 10ppb	16.79	49469					
modifier blank #2	ND	0					
c1c	12.01	36787	2.075	0.951	54.17%	0.120	0.13
c2a	2.00	7335	2.039	1.062	47.92%	0.020	0.02
c2b	2.74	9682	2.123	1.169	44.94%	0.027	0.02
c2c	0.80	3472	1.847	0.991	46.35%	0.008	0.01
c3a	601.64	1407493	1.498	0.670	55.27%	6.016	8.98
c3b	609.00	1424522	1.605	0.732	54.39%	6.090	8.32
c3c	449.45	1055426	1.908	0.848	55.56%	4.495	5.30
c4a	193.46	463252	1.768	0.782	55.77%	1.935	2.47
c4b	140.54	340821	1.388	0.576	58.50%	1.405	2.44
c4c	166.09	399920	1.440	0.574	60.14%	1.661	2.89
calibration check 100ppb	78.79	173327					
modifier blank #3	ND	0					
calibration check 10ppb	14.14	42528					

Calibration Checks	Actual	% difference
10 ug/L	16.79	67.9%
100 ug/L	78.79	-26.9%
10 ug/L	14.14	41.4%

Samples collected on Jan 28 2000, analyzed on 6 Feb 2000
50degC-50min in Tekmar 7000 using 100uL sample loop
Nitrogen purge and carrier gas
GC# 57244, range 10, sensitivity unknown, 70m x 0.45mm, 2.55 um film, DB-VRX column
Temps: GC injection - 200C, ECD - 300C, sample loop and transfer line - 150C
GC Program: 100C-8min, 40C/min to 220C, hold 1 min

* Calibrations:
TCE (ug/L) = 1E-09x2 + 0.0002x + 1.0235, R2 = 0.9993, using 2-100ppb standards

Jan 2000 c3 and c4 values were far above the highest standard. However, Mar 2000 calibrations/standards were nearly identical to Jan 2000, so Mar calibrations were used to estimate c3 and c4 values.
   TREES c3 and c4...TCE (ug/L) = 0.000432283 * AREA - 6.79197, r^2 = 0.9996, March 00 calibration

Table A.2 February 2000 tree-core TCE concentration data.

Sample Type	x (ppb) *	y (area)	wet core wt (g)	core length (cm)	dry core wt (g)	% water	mass TCE (ug)	ug/g (TCE in dry core)
blank		134						
modifier blank #1	0.52	64						
Avg of blanks	0.00	48						
2		323						
5		1847						
10		12251						
25		24150						
50		44102						
100		78136						
200		135048						
300		208823						
400		287851						
500		370987						
modifier blank #2	0.64	109						
600		145911						
modifier blank #3		43						
calibration check #1, 25ppb	268.63	195124						
modifier blank #4	0.44	35						
c5a	4.42	1516	1.879	9.500	0.806	57.10	0.044	0.0549
c5b	2.31	732	1.565	9.500	0.844	46.07	0.023	0.0274
c5c	0.04	1779	1.427	9.500	0.791	44.57	0.000	0.0004
c6a	2.88	3823	1.349	7.250	0.911	32.47	0.029	0.032
c6b	4.38	4906	1.835	8.750	1.264	31.12	0.044	0.035
c6c	5.57	5763	1.732	8.500	1.127	34.93	0.056	0.049
calibration check #2, 25ppb	146.83	107444						
modifier blank #5	0.48	50						
c7a	89.35	66071	1.723	9.500	0.907	47.36	0.893	0.99
c7b	69.11	51502	1.714	9.500	0.787	54.08	0.691	0.88
c7c	84.22	62375	1.750	9.000	0.815	53.43	0.842	1.03
c8a	0.48	50	2.012	9.500	0.821	59.19	0.005	0.0058
c8b	0.49	54	2.061	9.000	0.824	60.02	0.005	0.0059
c8c	0.44	36	2.225	10.000	0.968	56.49	0.004	0.0045
c9a	0.49	55	1.229	9.500	0.824	32.95	0.005	0.0060
c9b	0.49	55	1.198	9.500	0.785	34.47	0.005	0.0063
c9c	0.45	39	1.307	10.000	0.872	33.28	0.004	0.0051
calibration check #3, 25ppb	254.05	184628						
modifier blank #6	0.48	50						

Collected 28 feb 00, analyzed on 1 mar 00 after 1st run failed, all samples contained previously punctured septa, data is expected to be low.

50degC-50min in Tekmar 7000 using 100uL sample loop
GC 57244, range 10, sensitivity unknown, 70m x 0.45mm, 2.55 um film, DB-VRX column
GC Program: 100C-8min, 40C/min to 220C, hold 1 min

* Calibrations:
for AREA > 1753....TCE (ug/L) = (AREA - 1753.4)/719.84, r^2 = 0.9975, using standards from 2 to 500 ug/L
for AREA < 1753....TCE (ug/L) = (AREA + 127.5)/371.5, r^2 = 0.9308, using modifier blank average plus the 2 and 5 ug/L standards

Tree C6 is near well 064. Tree C9 is a control tree (russian olive) next to C8.

Calibration checks were made separate from the calibration standards and from different stock.....therefore, cal checks are not believed to be trustworthy.

Table A.3 March 2000 tree-core TCE concentration data.

Sample or Standard (ppb)	x (ppb) *	y (area)	wet core wt (g)	dry core wt (g)	dry wt fraction	mass TCE (ug)	ug/g (TCE in dry core)
			Head Space				
modifier blank #1	0.062	215					
2	3.11	10786					
5	5.79	20080					
10	10.29	35652					
25	24.64	85373					
50	51.69	135286					
100	104.56	257601					
200	203.54	486558					
300	296.25	701023					
400	394.21	927643					
500	457.37	1073746	not used				
600	684.47	1599091	averaged				
modifier blank #2	0.095	330					
modifier blank #3	0.019	65					
500	391.54	921459	not used				
600	522.04	1223348	averaged				
modifier blank #4	0.089	309					
c-8 #1 (control)	0.022	76	1.159	0.434	0.374	2.19E-04	5.05E-04
c-8 #2 (control)	0.021	74	1.013	0.367	0.362	2.14E-04	5.82E-04
c-8 #3 (control)	0.023	78	1.377	0.500	0.363	2.25E-04	4.50E-04
c-10 #1	541.73	1268907	1.096	0.440	0.401	5.417	12.31
c-10 #2	237.19	564404	0.889	0.455	0.512	2.372	5.21
c-10 #3	501.62	1176099	1.017	0.437	0.430	5.016	11.48
trip blank #1	0.057	199					
c-3 #1 spike 20ppb **	1042.98	2428441					
c-3 #2	469.21	1101137	1.061	0.399	0.376	4.692	11.76
c-3 #3 **	1159.33	2697582	0.990	0.388	0.392	11.593	29.88
c-11 #1	84.86	212024	1.021	0.402	0.394	0.849	2.11
c-11 #2 spike 20ppb	89.35	222406					
c-11 #3	77.52	195032	1.008	0.435	0.432	0.775	1.78
c-7 #1	101.03	249414	0.802	0.384	0.479	1.010	2.63
c-7 #2	72.85	184231	0.834	0.376	0.451	0.728	1.94
c-7 #3	120.56	294607	0.991	0.447	0.451	1.206	2.70
modifier blank #5	0.053	182					
calibration check (25ppb)	75.35	190021					

10 Mar 2000 OU4 samples:

50degC-50min in Tekmar 7000 using 100uL sample loop
GC Settings: Range=10, current=1, program: 100C-8min, 40C/min to 220C, hold 1 min
Column: J&W Science Cat no 1241574, DB-VRX, 2.55 micron, estimate length at 75m x .45mm

* Calibrations:
for AREA > 85374...TCE (ug/L) = 0.000432283 * AREA - 6.79197, r^2 = 0.9996, using 2 through 600 excluding those marked (equation generated by Class VP software)

for AREA < 85374...TCE (ug/L) = AREA/3465.3, r^2 = 0.9924, using standards 2 - 25 and forcing through zero

** estimates only. Above highest standard, assumed linear regression beyond highest standard.

Notes: % recovery (both spikes) not trustworthy because of extreme variability among the replicate samples, TCE/tissue sorption is expected to be the problem.

Used different TCE/methanol stock solution for calibration checks (stock for standards was different). Cal check is speculative.

Table A.4 April 2000 tree-core TCE concentration data.

Sample Type	Head Space		core wt (wet, g)	core length (cm)	core wt (dry, g)	dry wt fraction	mass TCE (ug)	ug/g (TCE in dry core)
	x (ppb) *	y (area)						
modifier blank #1	0	0						
2	1.78	16050						
10	10.04	93019						
50	49.24	204550						
100	110.44	353620						
300	296.87	807764						
modifier blank #2	0.08	306						
calibration check #1 (20ppb)	30.00	157688						
c-8 #1 (control)	ND	0	2.006	9.000	0.822	0.41	0.000	ND
c-8 #2 (control)	ND	0	1.958	9.000	0.814	0.42	0.000	ND
spike 20ppb c-8 #3	2.96	27096	2.057	9.500	0.833	0.40	0.030	0.04
c3#1	461.63	1209126	0.673	9.500	0.740	1.10	4.616	6.24
c3#2 split	209.48	594893	0.568	See note.	0.319	0.56	2.095	6.57
c3#3 split	206.10	586663	0.645	See note.	0.319	0.49	2.061	6.46
c4#1 split	331.44	891981	0.951	See note.	0.436	0.46	3.314	7.60
c4#2	436.78	1148594	2.241	9.000	0.826	0.37	4.368	5.29
c4#3 split	275.53	755796	0.896	See note.	0.424	0.47	2.755	6.50
modifier blank #3	0.06	114						
calibration check #2 (20ppb)	49.94	206247						
c10 #1	292.18	796353	1.412	9.000	0.758	0.54	2.922	3.85
c10 #2	209.31	594472	1.679	10.000	0.875	0.52	2.093	2.39
c10 #3	259.92	717759	1.594	10.000	0.821	0.52	2.599	3.17
c7#1 split	24.63	144588	0.979	See note.	0.440	0.45	0.246	0.56
c7#2 split	7.66	103269	0.739	See note.	0.369	0.50	0.077	0.21
c7#3	40.31	182798	1.807	10.000	0.818	0.45	0.403	0.49
c1#1	2.31	20991	1.798	10.500	0.956	0.53	0.023	0.02
c1#2	1.92	17368	1.695	10.000	0.904	0.53	0.019	0.02
spike 20ppb c1#3	4.61	42425	1.753	10.500	0.932	0.53	0.046	0.05
modifier blank #4	0.06	101						
calibration check #3 (20ppb)	68.40	251220						

13 Apr 00 samples:

50degC-50min in Tekmar 7000 using 100uL sample loop
GC Settings: Range=10, current=unknown, program: 100C-8min, 40C/min to 220C, hold 1 min
Column: J&W Science DB-VRX, 2.55 micron, length not recorded but estimate a 70m x 0.45mm

* Calibrations:
for AREA > 93020...TCE (ug/L) = (AREA - 84598)/2436, r^2 = 0.9967, using standards 10 to 300 ppb
for AREA < 93020...TCE (ug/L) = (AREA +477.03)/9308.8, r^2 = 0.9999, using zero, 2, and 10 ug/L

Notes: Split samples were cut up into small pieces using scissors, shaken to randomly distribute, then split roughly into two samples (1 in methanol, 1 in modifier); therefore, no core length exists for these samples.

Since calibration checks were made from different stock solutions than the calibration standards (an early mistake, there is no way to compare/verify values. Standards were made gravimetrically (same way they were made in both 2000 and 2001). The later months contain calibration checks that closely resemble the standards since they were made from the identical stock. Confidence is high in the standards but low in the calibration checks.   Spike recovery is poor. Expected cause is TCE/tissue sorption.

Table A.5 May 2000 tree-core TCE concentration data.

Sample Type	Head Space							
	x (ppb) *	y (area)	core wt (wet)	core length (cm)	core wt (dry)**	dry weight fraction	mass TCE (ug)	ug/g (TCE in dry core)
modifier blank	ND	0						
2	2.46	139						
26.6	26.57	1298						
80.7	84.83	3565						
155.5	153.17	6288						
269.1	270.75	8670						
400.4	399.62	11313						
543.6	448.09	12307						
modifier blank	ND	0						
calibration check 50ppb	281.77	8896						
modifier blank	ND	0						
C-1 #1	1.07	72	1.646	11.000	0.849	0.516	0.011	0.013
C-1 #2	1.11	74	1.689	11.000	0.872	0.516	0.011	0.013
calibration check #1 (50 PPB)	288.35	9031						
Blank	ND	0						
C-1 SPK (40 PPB)	14.48	717	1.660	10.500	0.857	0.516		
C-2#1	0.63	51	2.030	10.000	1.066	0.525	0.006	0.006
C-2#2	0.51	45	1.792	10.000	0.941	0.525	0.005	0.005
C-2#3	0.49	44	1.933	10.000	1.015	0.525	0.005	0.005
C-8#1 RO	13.07	649	2.117	10.000	0.881	0.416	0.131	0.148
C-8#2 RO	ND	0	2.056	10.000	0.855	0.416	ND	ND
C-8#3 SPK (40 PPB)	ND	0	2.106	10.500	0.876	0.416	ND	ND
C-3#1	317.27	9624	1.626	10.000	0.800	0.492	3.173	3.97
C-3#2	443.45	12212	1.638	10.000	0.806	0.492	4.435	5.50
C-3#3	370.22	10710	1.539	10.500	0.757	0.492	3.702	4.89
Blank	ND	0						
calibration check #2 50 PPB	223.26	7696						
Blank	ND	0						
C-4#1 RO	357.35	10446	1.751	8.500	0.763	0.436	3.573	4.68
C-4#2 RO	264.26	8537	2.192	10.000	0.956	0.436	2.643	2.77
C-4#3 RO	316.44	9607	2.018	9.000	0.880	0.436	3.164	3.60
C-7#1 POP	37.55	1681	1.885	10.500	0.903	0.479	0.376	0.42
C-7#2 POP	33.76	1530	1.839	10.000	0.881	0.479	0.338	0.38
C-7 SPK (40 PPB)	50.65	2203	1.897	10.000	0.909	0.479	0.507	0.56
C-11#1 POP	25.61	1252	1.870	10.000	0.896	0.479	0.256	0.29
C-11#2 POP	24.38	1193	1.924	10.000	0.922	0.479	0.244	0.26
C-11#3 POP	25.94	1268	1.908	10.000	0.914	0.479	0.259	0.28
C-10#1 POP	153.17	6288	2.072	10.000	0.992	0.479	1.532	1.54
Blank	ND	0						
calibration check #3 50 PPB	276.50	8788						
Blank	ND	0						
C-10#2 POP	167.14	6545	2.174	11.000	1.041	0.479	1.671	1.61
C-10#3 POP	182.01	6850	2.099	10.500	1.005	0.479	1.820	1.81

Collected on 18 May 00, analyzed on 31 May 00:

50degC-50min in Tekmar 7000 using 100uL sample loop and nitrogen carrier gas
GC #50220 Settings: program: 100degC-8min, 40degC/min to 220degC, hold 1 min. Inject temp = 200C
Detector Settings: Range=10, current=1, temp = 300C, total nitrogen flow = 50mL/min
Column: DB-VRX, 2.55 micron, 75m (estimate) x 0.45mm
Samples collected between late morning to early afternoon on southern side, sunny day, spikes* were 40ml of 9.92ppm std (39.68ppb in 10ml of modifier)

* Calibrations: (best fit to calibration data required 3 separate linear regressions)
for AREA >6289.....TCE (ppb) = (AREA - 3117.2)/20.509, r^2 = 0.9999, used standards 155.5 to 400.4ppb
for AREA < 6289 but > 1299....TCE (ppb) = (AREA - 184.5)/39.849, r^2 = 0.9975, used standards 2 to 155.5ppb
for AREA < 1299....TCE (ppb) = (AREA - 20.577)/48.086, r^2 = 0.9991, used standards 2 and 26.6ppb and forced through zero

** estimated using average dry weight fractions from Jan-Aug '00

Notes: Results were similar to months previous and after with exception to high June results.

Again, poor spike recovery (due to sorption) and calibration checks. However, replicate calibration checks were very similar and detector drift is minimal to none. Still unwisely using a different stock TCE/methanol solution to make the calibration checks. Standards were made gravimetrically and were new, calibration check solution was several weeks old but kept in freezer (may have been reason for poor calibration check results).

Table A.6 June 2000 tree-core TCE concentration data using pH 2 modifier solution.

Site	Head Space							
	y (area)	x (ug/L) polyfit *	core wt (wet)	core length (cm)	core wt (dry) calculated	dry wt fraction	mass TCE (ug) (x polyfit * 0.01L)	ug/g (TCE in dry core)
pH 2 Samples								
ph2 blank	0	0.00						
10.01	0	not used						
48.44	1647	45.90						
93.98	3051	128.92						
475.2	9854	546.03						
1003	16160	957.77						
5172	56958	5098.81						
8705	81437	not used						
ph2 blank	0	0.00						
cal chk 986 ppb	28362	1844.74						
ph2 blank	0	0.00						
2-1 (c-10 poplar core)	20883	1285.11	1.779	9.500	0.904	0.508	12.851	14.22
2-2 (c-10 poplar core)	20111	1230.36	1.866	10.000	0.948	0.508	12.304	12.98
2-3 (c-10 poplar core)	20347	1247.04	1.711	9.500	0.869	0.508	12.470	14.35
2-4 (c-10 poplar core)	19338	1176.05	1.807	10.000	0.918	0.508	11.760	12.81
2-5 (c-10 poplar core)	21498	1329.09	1.706	9.500	0.867	0.508	13.291	15.34
2-6 (c-10 poplar core)	21007	1293.95	1.709	9.500	0.868	0.508	12.939	14.90
2-7 (c-3 cotton shav)	20961	1290.67	1.565	did not measur	0.817	0.522	12.907	15.80
2-8 (c-3 cotton shav)	15824	935.14	1.588	did not measur	0.829	0.522	9.351	11.28
2-9 (c-3 cotton shav)	20195	1236.29	1.735	did not measur	0.906	0.522	12.363	13.65
2-10 (c-4 R.O. shav)	6320	326.16	1.532	did not measur	0.614	0.401	3.262	5.31
2-11 (c-4 R.O. shav)	5073	250.25	2.036	did not measur	0.816	0.401	2.503	3.07
2-12 (c-4 R.O. shav)	4997	245.66	2.211	did not measur	0.887	0.401	2.457	2.77
2-13 (c-3 cotton core)	35874	2474.01	1.385	10.000	0.723	0.522	24.740	34.22
2-14 (c-3 cotton core)	37121	2586.89	1.525	10.000	0.796	0.522	25.869	32.50
2-15 (c-3 cotton core)	32957	2220.08	1.434	10.000	0.749	0.522	22.201	29.66
2-16 (c-10 poplar shav)	4595	221.38	2.038	did not measur	1.035	0.508	2.214	2.14
ph2 blank	0	0.00						
cal chk 986 ppb	27730	1795.17						
ph2 blank	0	0.00						
2-17 (c-10 poplar shav)	3932	181.53	2.396	did not measur	1.217	0.508	1.815	1.49
2-18 (c-10 poplar shav)	1977	65.32	1.621	did not measur	0.823	0.508	0.653	0.79
2-19 (c-4 R.O core)	19112	1160.26	1.452	6.500	0.582	0.401	11.603	19.93
2-20 (c-4 R.O core)	15236	895.74	1.628	7.500	0.653	0.401	8.957	13.72
2-21 (c-4 R.O core)	20638	1267.68	1.857	8.500	0.745	0.401	12.677	17.02
ph2 blank	0	0.00						
cal chk 986 ppb	29157	1907.75						
ph2 blank	0	0.00						

8 Jun 00 samples using:

50degC-50min in Tekmar 7000 using 100uL sample loop
GC Settings: Range=10, current=1, program: 100C-8min, 40C/min to 220C, hold 1 min
Column: J&W Science Cat no 1241574, DB-VRX, 2.55 micron, 30m x 0.45mm
Detector Temp 300C, Inj Temp 200C, transfer line temp 150C, sample loop temp 150C

* Calibrations:
ph2: AREA = -0.0012(x^2) + 17.12x + 863.79, R^2 = 0.9987
ph10: AREA = -0.0011(x^2) + 14.475x + 1847.3, R^2 = 0.9994
core (no modifier): TCE (ug) = (AREA - 26350)/900000000

Notes: Calibration checks not trustworthy (although replicates very similar), used different stock to make cal chk standards.

pH2 Poly curve using 10 to 5172 ppb stds

$y = -0.0012x^2 + 17.12x + 883.79$
$R^2 = 0.9987$

(x-axis: TCE µg/L, y-axis: area)
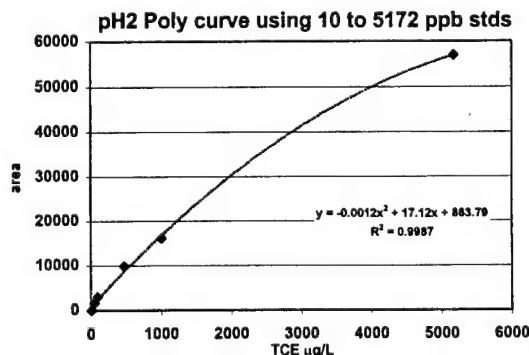
Table A.7 June 2000 tree-core TCE concentration data using pH 10 modifier solution.

| Site | Head Space | | | | | | | |
	y (area)	x (ug/L) polyfit *	core wt (wet)	core length (cm)	core wt (dry) calculated	dry wt fraction	mass TCE (ug) (x polyfit * 0.01L)	ug/g (TCE in dry core)
pH 10 Samples								
ph10 blank	0	0.00						
10.01	0.00	0.00						
48.44	1619.00	not used						
93.98	2824.00	67.82						
475.20	9173.00	527.22						
1003.00	14927.00	975.99						
5172.00	47212.00	5147.84						
8705.00	86600.00	not used						
ph10 blank	0	0.00						
ph10 CC 986ppb	27331	2093.63						
ph10 blank	0	0.00						
10-1 (c-10 poplar core)	16460	1101.76	1.618	9.000	0.822	0.508	11.018	13.40
10-2 (c-10 poplar core)	16418	1098.27	1.633	9.500	0.830	0.508	10.983	13.24
10-3 (c-10 poplar core)	17164	1160.49	1.646	9.500	0.836	0.508	11.605	13.88
10-4 (c-3 cotton core)	32754	2681.67	1.476	10.000	0.770	0.522	26.817	34.81
10-5 (c-3 cotton core)	31140	2497.79	1.469	10.000	0.767	0.522	24.978	32.57
10-6 (c-3 cotton core)	33488	2768.23	1.343	9.000	0.701	0.522	27.682	39.49
10-7 (c-4 R.O core)	15766	1044.47	2.104	9.500	0.844	0.401	10.445	12.38
10-8 (c-4 R.O core)	18109	1240.35	1.904	9.000	0.764	0.401	12.403	16.25
10-9 (c-4 R.O core)	16434	1099.60	1.878	8.500	0.753	0.401	10.996	14.60
ph10 blank	0	0.00						
ph10 CC 986ppb	28555	2219.42						
ph10 blank	0	0.00						

Table A.8 June 2000 tree-core TCE concentration data using NO modifier solution.

Sample Type	TCE (ug)	y (area)	wet core wt (g)	core length (cm)	core volume (mL)	HS volume (mL)	dry core wt (g)	dry wt fraction	mass TCE (ug)	ug/g (TCE in dry core)
Core Only (no modifier)										
1.73	1.73E-06	8261								
4.95	4.95E-06	17786								
9.94	9.94E-06	28382								
24.68	2.47E-05	39397								
51.27	5.13E-05	86822								
98.28	9.83E-05	131060								
260.43	2.60E-04	297600								
500.39	5.00E-04	548390								
1319.15	1.32E-03	1265863								
empty blank	ND	0								
E-1 (c-10 poplar hi core)	1.88E-05	36228	1.730	10.000	2.027	20.253	0.901	0.521	3.80E-01	4.22E-01
E-2 (c-10 poplar hi core)	1.94E-05	36978	1.764	10.000	2.027	20.253	0.863	0.489	3.93E-01	4.55E-01
E-3 (c-10 poplar hi core)	1.73E-05	34539	1.710	10.000	2.027	20.253	0.880	0.515	3.50E-01	3.98E-01
E-4 (c-3 cotton hi core)	4.32E-05	64358	1.374	9.500	1.925	20.355	0.739	0.538	8.78E-01	1.19E+00
E-5 (c-3 cotton hi core)	4.00E-05	60773	1.471	10.000	2.027	20.253	0.751	0.511	8.11E-01	1.08E+00
E-6 (c-3 cotton hi core)	4.33E-05	64530	1.565	10.000	2.027	20.253	0.811	0.518	8.77E-01	1.08E+00
E-7 (c-4 R.O hi core)	7.80E-06	23495	1.818	8.500	1.723	20.557	0.751	0.413	1.60E-01	2.14E-01
E-8 (c-4 R.O hi core)	8.28E-06	24053	1.843	8.000	1.621	20.659	0.717	0.389	1.71E-01	2.39E-01
E-9 (c-4 R.O hi core)	6.53E-06	22012	1.807	8.500	1.723	20.557	0.723	0.400	1.34E-01	1.86E-01
E-10 (ou2 pop cntrl core)	ND	0	1.915	10.000	2.027	20.253	0.795	0.415	ND	ND
E-11 (ou2 pop cntrl core)	ND	0	1.765	10.000	2.027	20.253	0.784	0.444	ND	ND
E-12 (ou2 pop cntrl core)	ND	0	1.882	10.000	2.027	20.253	0.779	0.414	ND	ND
empty blank	ND	0								

7-13-00 Direct Injection Cal Curve (area vs mass on detector), 1μL direct injection of std, GC#57244 containing 30m x0.45mm 2.55 micron DB-VRX column. GC settings: range = 10, current = 1, program: 45c-2min, 5c/min to 70c, then 40c/min to 100c, 40c/min to 220c, hold 1 min....det temp 300c, inj temp 200c

Calibration: Class VP software generated equation. TCE (ug) = 1.58526e-16*Area^2 + 8.51291e-10*Area-1.22878e-5

Notes: Actual mass extracted by placing cores into empty headspace vials is only an estimate. Although the headspace temp program is the same, the amount of headspace drawn into the sample loop is different because the empty headspace vials will be at a different static pressure at 50c.

Detector Sensitivity to TCE Using Direct Injections (1μL)
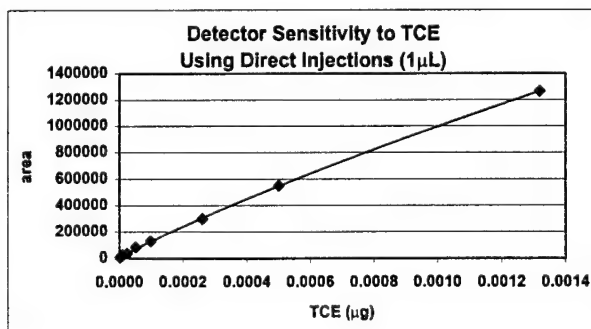
Table A.9 July 2000 tree-core TCE concentration data.

Sample Type	Head Space							
	x (ppb) *	y (area)	wet core wt (g)	core length (cm)	calculated dry core wt (g)	dry wt fraction	mass TCE (ug)	ug/g (TCE in dry core)
1	1.18	575						
5	4.83	2393						
10	10.33	5133						
52	50.53	15667						
103	108.73	29700						
503	502.52	124656						
1015	1156.11	282255						
5106	4006.71	969622						
modifier blank #1	0.99	477						
calibration check 503.49	598.26	147742						
modifier blank #2	0.55	261						
c-1 (box elder)	0.66	313.00	1.577	10.000	0.906	0.575	0.007	0.007
c-1 (box elder)	0.80	386.00	1.568	10.000	0.901	0.575	0.008	0.009
c-1 (box elder)	1.04	502.00	1.724	10.000	0.991	0.575	0.010	0.010
c-2 (apple)	0.97	468.00	1.904	10.000	1.159	0.609	0.010	0.008
c-2 (apple)	ND	0.00	1.831	10.000	1.114	0.609	ND	ND
c-2 (apple)	0.45	208.00	1.892	10.500	1.151	0.609	0.004	0.004
c-8 (R.O. control)	0.85	407.00	2.077	10.000	1.023	0.493	0.008	0.008
c-8 (R.O. control)	0.68	325.00	1.943	9.000	0.957	0.493	0.007	0.007
c-8 (R.O. control) spike	4.75	2354.00	2.023	9.000	0.996	0.493	0.048	0.048
c-4 (R.O.)	171.19	44761.00	2.037	10.000	1.003	0.493	1.712	1.71
modifier blank #3	0.40	183.00						
calibration check 102.96	126.91	34084.00						
modifier blank #4	0.40	182.00						
c-4 (R.O.)	218.27	56114.00	2.217	10.500	1.092	0.493	2.183	2.00
c-4 (R.O.)	139.00	36999.00	1.945	9.000	0.958	0.493	1.390	1.45
c-3 (cotton)	250.66	63925.00	1.655	10.500	0.953	0.576	2.507	2.63
c-3 (cotton)	231.76	59367.00	1.467	10.000	0.845	0.576	2.318	2.74
c-3 (cotton)	253.84	64692.00	1.740	10.000	1.002	0.576	2.538	2.53
c-10 (poplar)	204.64	52829.00	1.829	10.500	1.099	0.601	2.046	1.86
c-10 (poplar)	226.57	58115.00	1.870	11.000	1.124	0.601	2.266	2.02
c-10 (poplar) spike	216.54	55697.00	1.550	10.500	0.931	0.601	2.165	2.33
c-7 (poplar)	23.19	9075.00	1.993	10.000	1.024	0.514	0.232	0.226
c-7 (poplar)	21.65	8703.00	2.000	10.000	1.028	0.514	0.216	0.211
c-7 (poplar)	21.37	8636.00	1.908	10.000	0.981	0.514	0.214	0.218
modifier blank #5	ND	0.00						
calibration check 52	46.72	14748.00						
modifier blank #6	0.51	239.00						

Collected on 31 July 00, analyzed on 8/7/00:

50degC-50min using Tekmar 7000 and 100uL sample loop, transfer line/loop temp = 150C
GC #57244 Settings: Range=10, current=1, program: 100degC-8min, 40degC/min to 220degC, hold 1 min
Column: J&W Science Cat no 1241574, DB-VRX, 2.55 micron, unsure of length
Samples collected on North Side of trees between 1400hrs and 1630hrs, sunny day, spikes were 20ml of 25.826ppm std (51.7ppb in 10ml of modifier)

* Calibrations:
for AREA < 5134....TCE (ppb) = (AREA + 14.787)/498.19, r^2 = 0.996, used standards 1 to 10.18ppb
for AREA > 5133.....TCE (ppb) = (AREA - 3483)/241.13, r^2 = 0.9997, used standards 10.18 to 503.49ppb

Notes: Dry weights calculated using dry weight averages from previous months.

Table A.10 August 2000 tree-core TCE concentration data.

Sample Type	Head Space		wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
	x (ppb) *	y (area)					
7.56	6.04	312					
15.11	16.95	689					
59.82	59.47	2159					
149.55	182.03	5274					
511.1	455.64	10414					
1022.19	1045.18	21489					
2044.39	2331.35	45651					
C-8 (R.O.)#1	0.00	0.00	2.040	9.500	0.000	ND	ND
C-8 (R.O.)#2	0.00	0.00	2.093	10.000	0.000	ND	ND
C-3 (Cotton)#1	461.98	10533.00	1.659	10.000	0.845	4.620	5.47
C-3 (Cotton)#2	446.97	10251.00	1.330	10.000	0.677	4.470	6.60.
C-3 (Cotton)#3 spiked	261.14	6760.00	1.696	10.000	0.863	2.611	3.02
C-4 (R.O.)#1	135.15	4775.00	2.088	9.500	0.939	1.351	1.44
C-4 (R.O.)#2	131.04	4633.00	2.189	9.500	0.984	1.310	1.33
C-10 (Poplar)#1	133.50	4718.00	1.323	10.000	0.797	1.335	1.67
C-10 (Poplar)#2	140.82	4971.00	1.433	9.500	0.863	1.408	1.63
C-7 (Poplar)#1	29.65	1128.00	1.698	10.500	0.959	0.296	0.31
C-7 (Poplar)#2	33.09	1247.00	1.797	10.000	1.015	0.331	0.33
C-11 (Poplar)#1	12.93	550.00	1.897	10.000	0.963	0.129	0.13
C-11 (Poplar)#2	13.50	570.00	1.911	10.000	0.970	0.135	0.14
C-11 (Poplar)#3 spiked	50.21	1839.00	2.097	10.500	1.065	0.502	0.47
C-1 (Box Elder)#1	0.00	0.00	1.561	9.500	0.922	ND	ND
C-1 (Box Elder)#2	0.00	0.00	1.578	10.000	0.932	ND	ND
C-2 (Apple)#1	0.00	0.00	1.937	10.000	1.240	ND	ND
C-2 (Apple)#2	0.00	0.00	1.785	10.000	1.142	ND	ND
No modifier used below C-8 (R.O.)	ND	0.00	1.912	9.500	0.867	ND	ND
C-3 (Cotton)	1.44	97779.00	1.538	10.000	0.783	0.020	0.02614
C-4 (R.O.)	0.35	51928.00	2.458	11.000	1.105	0.005	0.00424
C-10 (Poplar)	0.24	47223.00	1.557	10.500	0.938	0.003	0.00350
C-7 (Poplar)	0.009	17173.00	1.676	10.000	0.947	0.000	0.00013
C-11 (Poplar)	0.005	9673.00	1.920	10.000	0.975	0.000	0.00007
C-1 (Box Elder)	ND	0.00	1.660	10.500	0.980	ND	ND
C-2 (Apple)	ND	0.00	1.779	9.500	1.139	ND	ND

Collected on 30 Aug 00, analyzed on 9/4/00:

Tekmar 7000 50C for 50 min equilibration time using 100ul sample loop at 150C, transfer line at 150C, mix power 8 for 5min,
    carrier gas flow ~12mL/min nitrogen, detector total flow = ~52mL
GC#57244, 100C-8min, 40c/min to 220c, hold 1min, injection temp =200c
Detector settings: range=10, current=0.5 , temp = 300c
Column: DB-VRX 75m x 0.45mm column, 2.55 micron film thickness

Samples collected between 1500hrs and 1800hrs, sunny day, spikes* were 50ul of 29.91ppm std (149.55ppb in 10ml of modifier)

* Calibrations:
for area counts < 5274, ppb = (AREA - 103.16)/34.569 , r^2 = 0.9995, (cal range 7.56 to 149.55ppb)
for area counts > 5274, ppb = (AREA - 1854.3)/18.786, r^2 = 0.988, (cal range 149.55 to 1022.19ppb)
linear formula for direct injection: AREA > 37391, ug/L = (.0023852xAREA)-88.8069, r^2=0.999327, range (1.43-2033ppb)
linear formula for direct injection: AREA < 37392, ug/L = (AREA + 4E-12)/19418, r^2=1, range (0 to 1.43ppb), two point curve only for lowest results

Table A.11 Nocturnal sampling results, 9/15/00

Time	Mean TCE (mg/kg)	SD (mg/kg)	Range (mg/kg)
20:45	1.92	0.22	0.26 - 2.07
1:40	2.36	0.17	2.23 - 2.56
5:45	2.19	0.10	2.09 - 2.28
9:20	3.76	0.93	2.82 - 4.67
13:05	2.71	0.24	2.47 - 2.94

Table A.12 October 2000 tree-core TCE concentration data.

Site	Head Space						
	x (ppb) *	y (area)	wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
modifier blank #1	ND	0					
0.27	0.40	5758					
5.72	5.56	57092					
28.59	28.62	286913					
108.79	107.45	811836					
435.16	435.82	2461260					
870.31	870.11	4045213					
modifier blank #2	ND	0					
c1a	0.82	9928	1.703	10.000	0.852	0.008	0.010
c1b	0.50	6698	1.679	10.500	0.839	0.005	0.006
c2a	0.36	5312	1.877	9.500	0.938	0.004	0.004
c2b	0.13	3034	1.942	9.500	0.971	0.001	0.001
c8a	ND	0	2.085	10.000	1.043	ND	ND
c8b	ND	0	2.008	10.000	1.004	ND	ND
c3a	472.18	2612194	1.559	10.000	0.780	4.722	6.06
c3b	587.37	3064088	1.557	9.500	0.778	5.874	7.54
c4a	253.87	1626672	1.847	9.000	0.923	2.539	2.75
c4b	188.33	1282882	2.029	9.000	1.015	1.883	1.86
modifier blank #3	ND	0					
cal chk #1 108.79	81.07	643657					
modifier blank #4	ND	0					
c10a	139.79	1007435	1.447	9.500	0.724	1.398	1.93
c10b	148.71	1059588	1.511	9.500	0.756	1.487	1.97
c7a	33.72	317985	2.066	10.500	1.033	0.337	0.33
c7b	31.97	305220	1.906	10.500	0.953	0.320	0.34
c11a	26.89	269652	1.993	10.000	0.996	0.269	0.27
c11b	30.34	293343	2.060	10.000	1.030	0.303	0.29
c20a	ND	0	1.732	11.000	0.866	ND	ND
c20b	ND	0	1.794	11.000	0.897	ND	ND
c21a	1.29	14627	1.986	10.500	0.993	0.013	0.01
c21b	0.90	10683	2.013	10.500	1.007	0.009	0.01
modifier blank #5	ND	0					
cal chk #2 108.79	97.35	748396					
modifier blank #6	ND	0					
c22a	9.16	93050	1.656	8.500	0.828	0.092	0.11
c22b	10.45	105875	1.617	9.500	0.809	0.105	0.13
trip blank 1	ND	0					
trip blank 2	ND	0					
modifier blank #7	ND	0					
cal chk #3 108.79	100.71	769621					
modifier blank #8	ND	0					

Collected on 2 Oct 00, analyzed on 10 Oct 00:

Tekmar 7000 Headspace Analyzer: 1mL sample loop at 150C, transfer line at 150C
GC #57244 Settings: Range=10, current=0.5, program: 100C-8min, 40C/min-220C, hold 1min
Column flow: 12mL/min nitrogen, purge flow 2mL/min, total ECD flow ~50mL/min
Column: J&W Science DB-VRX, 2.55 micron, 75m x 0.45mm
Samples collected between 1200hrs and 1500hrs, sunny day

* Calibrations:
AREA < 287000 ... TCE (ppb) = (AREA - 1742.4)/9963.4, r^2 = 0.9999, used standards 0.27 to 28.59ppb for regression
AREA > 287000 ... TCE (ppb) = (2.32282e-11*AREA^2) + (0.000123051*AREA) - 7.75423, r^2 = 0.9999, used 28.59 to 870.31ppb

Notes: C20 is 255 deg from well U4-080. C21 and 22 (218 and 178 degrees from U4-080) were one time samples from trees on a ridge near the secondary 'hot spot' near the old gate on the north-northwest edge of OU4.

Table A.13 November 2000 tree-core TCE concentration data

(1st set, low conc. samples)

Sample Type	x (ppb) *	y (area)	Head Space wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
0		0					
0.29		34611					
1.17		75359					
15.25		827770					
117.72		4161665					
453.33		10477368					
906.66		13102792					
1133.33		13488560	not used				
mod blk 2	0.03	4512					
c-1a	0.11	14672	1.791	10.0	0.896	0.00112	0.00126
c-1b	0.10	13305	1.746	10.0	0.873	0.00101	0.00116
c-1c	0.21	26353	1.711	10.0	0.855	0.00212	0.00248
c-2a	0.14	17780	2.061	10.0	1.031	0.00138	0.00134
c-2b	0.10	13740	1.914	10.0	0.957	0.00105	0.00110
c-2c	0.12	15512	2.094	10.0	1.047	0.00119	0.00114
cal chk 1 117.72ppb	84.23	3158672					
cherry-crabapple a	0.01	1544	2.819				
cherry-crabapple b	0.02	2224	3.298	Although measurable, results between these bars were			
cherry-crabapple c	0.01	1175	2.156	treated as below quantifiable limits (BQL) because			
apple w/skin a	0.01	1930	1.571	their area counts were below background as shown by			
apple w/skin b	0.01	1804	1.220	the results of the blanks.			
apple w/o skin	0.01	1943	1.363				
evergreen a	0.02	2296	1.395	10.0	0.698	0.00017	0.00024
evergreen b	0.02	2705	1.281	10.5	0.640	0.00020	0.00031
evergreen c	0.02	2544	1.095	10.0	0.547	0.00019	0.00034
mod blk 3	0.03	3958					

Collected on 2 Nov 00, analyzed on 11/9/00:

Tekmar 7000 Headspace Analyzer: 1mL sample loop at 150C, transfer line at 150C flowing 12mL/min nitrogen,
    50C for 50 min equil time, mix power 8 for 5 min
GC #57244 Settings: Range=10, current=0.5, program: 40C-1min,15C/min-145C, hold 1min,
    40C/min-220C, hold 1min
Column: J&W Science DB-VRX, 2.55 micron, 75m x 0.45mm
Samples collected between 1600hrs and 1800hrs, sunny day

* Calibrations:
1st Set: AREA > 75359....AREA = 0.0166*TCE^3-41.045*TCE^2+37729*TCE+261885, r^2 = 1, used 15 to 906 ppb
          AREA < 75360... AREA = -62431*TCE^2+137453, r^2 = 1, used 0.29 and 1.17 fitted through zero
2nd Set: AREA > 65633... AREA = -1968*TCE^2 = 31487*TCE + 67948, R^2 = 0.9999, using 1.17 to 906ppb
          AREA < 65634... AREA = -196.87*TCE^2 + 53481*TCE - 3E-06, r^2 = 1, using zero, 1.17 and 117ppb

Table A.14 November 2000 tree-core TCE concentration data

(2nd set samples)

2nd Set	x (ppb) *	y (area)	wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
0	0.00	0					
1.17	1.17	62303					
117.72	120.17	3567574					
453.33	451.19	10268213					
906.66	694.80	12444650					
1133.33	870.09	13337869	not used				
mod blk 4	0.07	3564					
bigtreebyshed a	1.07	101555	1.632	9.0	0.816	0.01068	0.013
bigtreebyshed b	0.15	72636	1.587	9.5	0.794	0.00149	0.002
bigtreebyshed c	1.23	65633	1.602	9.0	0.801	0.01233	0.015
appletreebyshed a	0.96	51097	2.171	9.5	1.086	0.00959	0.009
appletreebyshed b	1.02	54321	1.984	9.0	0.992	0.01020	0.010
appletreebyshed c	0.93	49375	2.054	9.0	1.027	0.00926	0.009
mod blk 5	0.04	2008					
cal chk 2 117.72ppb	103.24	3108927					
mod blk 6	0.04	2206					
apple#1shed a	0.04	2318	1.857	na	0.929	0.00043	0.00047
apple#1shed b	0.05	2856	1.734	na	0.867	0.00053	0.00062
apple#2shed a	0.05	2795	2.020	na	1.010	0.00052	0.00052
apple#2shed b	0.05	2809	2.415	na	1.208	0.00053	0.00044
mod blk 7	0.04	2004					

Notes: Evergreen is large evergreen between OU4 shed and South Weber Drive. Apple trees were between OU4 shed and home due west along S. Weber Drive. Fruits were collected from the same trees between the home and the shed. Big tree by shed is the largest tree (unknown species) between the OU4 shed and South Weber Drive. It appears that some carryover was affecting the results of the blanks - split peaks were identified in the chromatograms. A cracked GC inlet liner was later found to be causing the carryover and split peaks.

Table A.15 November 2000 tree-core TCE concentration data (1ˢᵗ set, high conc. samples)

Sample Type	x (ppb)	y (area)	wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
			Head Space				
mod blk #1	ND	0					
0.04	0.03	4484					
0.27	0.62	20562					
14.29	15.55	428315					
101.38	106.15	2902739					
561.26	561.26	10252583	saturated detector				
1123			saturated detector				
mod blk #2	0.04	3379					
c-8a	0.01	1502	2.000	9.5	1.000	0.000	0.00011
c-8b	0.01	1282	1.874	9.5	0.937	0.000	0.00008
c-8c	0.01	1071	2.053	10.0	1.027	0.000	0.00005
c-3a	507.49	9718619	1.666	9.5	0.833	5.600	6.72
c-3b	538.04	10027450	1.595	10.0	0.797	5.600	7.02
c-3c	560.92	10249374	1.908	10.0	0.954	5.600	5.87
cal chk 1a 101.38	120.94	3306611					
c-4a	425.23	8804060	2.035	9.0	1.018	5.600	5.50
c-4b	345.71	7770418	2.269	10.0	1.135	5.600	4.94
c-4c	389.74	8363983	2.157	9.5	1.079	5.600	5.19
c-10a	321.18	7413091	1.622	10.0	0.811	3.212	3.96
c-10b	249.35	6229379	1.561	9.5	0.781	2.494	3.19
c-10c	175.70	4736675	1.571	9.5	0.785	1.757	2.24
mod blk #3	0.01	1619					
0.04	0.05	5035					
0.27	0.52	17782					
14.29	12.31	339872					
101.38	96.71	2644797					
561.26	658.77	11122081	saturated detector				
1123			saturated detector				
mod blk #4	0.03	3202					
c-7 1a South	43.00	1177995	1.852	10.0	0.926	0.430	0.46
c-7 1b South	41.53	1137840	1.822	10.0	0.911	0.415	0.46
c-7 1c South	50.11	1372211	2.351	10.0	1.176	0.501	0.43
c-7 2a NorthWest	59.21	1620722	1.952	10.0	0.976	0.592	0.61
c-7 2b NorthWest	64.67	1769761	1.893	10.0	0.947	0.647	0.68
c-7 2c NorthWest	48.54	1329397	1.974	10.5	0.987	0.485	0.49
c-7 3a NorthEast	29.34	804926	1.941	10.0	0.970	0.293	0.30
c-7 3b NorthEast	26.35	723129	1.867	9.5	0.934	0.263	0.28
c-7 3c NorthEast	24.50	672689	1.879	10.0	0.940	0.245	0.26
cal chk 1b 101.38	81.04	2216916					
mod blk #5	0.01	1122					
c-11a	30.58	838642	2.004	9.5	1.002	0.306	0.31
c-11b	30.27	830173	1.993	10.0	0.997	0.303	0.30
c-11c	26.88	737648	1.912	9.5	0.956	0.269	0.28
c-12a	31.14	854029	1.763	9.5	0.881	0.311	0.35
c-12b	30.10	825671	1.775	10.0	0.887	0.301	0.34
c-12c	28.61	784899	1.825	9.5	0.913	0.286	0.31
mod blk #6	0.01	1097					
cal chk 2b 101.38	108.48	2966243					
mod blk #7	0.03	2809					

Collected on 2 Nov 00, analyzed on 11/14/00:

GC #57244 Settings: Range=10, current=0.5, program: 40C-1min,15C/min-145C, hold 1min, 40C/min-220C, hold 1min

Tekmar 7000 Headspace Analyzer: 100uL sample loop at 150C, transfer line at 150C flowing 12mL/min nitrogen, 50C for 50 min equil time, mix power 8 for 5 min

Column: J&W Science DB-VRX, 2.55 micron, length not recorded

Samples collected between 1600hrs and 1800hrs, sunny day

* Calibrations:

IF 3380 < AREA < 4736674, ppb = (AREA-3540.6)/27312, linear regression from average of 0.04, 0.27, 14.29, & 101.38 ppb standards, r^2 = 1

IF AREA > 4736674, ppb = 2.2468e-18 * AREA^3 - 7.66576e-12 8 AREA^2 + 4.06437e-5 * AREA - 0.32577, used all but 1123ppb. R^2 = 1

IF AREA < 3380, ppb = (AREA-700.83)/74011, r^2 = .9949, used zero, .04, and .27 ppb standards.

Table A.16 January 2001 tree-core TCE concentration data (low conc. samples)

Sample Type	x (ppb) *	y (area)	Head Space wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
hs-jan28low samples							
modifier blank 1	0.00	0					
0.1	0.10	14737					
0.1	0.10	16217					
0.1	0.10	16651					
1	1.00	123537					
1	1.00	132819					
1	1.00	125388					
21.37	21.37	1615756					
21.37	21.37	1322582					
21.37	21.37	1559365					
49.87	49.87	2932084					
49.87	49.87	3064572					
49.87	49.87	2601755					
71.25	71.25	6313318					
71.25	71.25	4961045					
71.25	71.25	3820821					
106.87	106.87	saturated					
106.87	106.87	saturated					
106.87	106.87	saturated					
modifier blank 2	0.011	1421					
modifier blank 3	0.006	780					
modifier blank 4	0.003	382					
c1a	0.03	4300	1.679	10.000	0.914	0.000	0.000369
c1b	0.03	3484	1.804	10.500	0.933	0.000	0.000293
c1c	0.04	5055	1.886	10.500	0.929	0.000	0.000427
c2a	0.12	15740	2.070	9.500	1.061	0.001	0.00116
c2b	0.13	17176	2.222	10.500	1.180	0.001	0.00114
c2c	0.16	20353	2.229	10.500	1.157	0.002	0.00138
c8a	ND	0	1.885	9.500	0.758	ND	ND
c8b	ND	0	1.770	10.000	0.715	ND	ND
c8c	ND	0	2.041	10.000	0.853	ND	ND
modifier blank 5	0.002	229					
cal chk #1 21.37ppb	19.46	1390021					
cal chk #2 21.37ppb	21.71	1547953					
modifier blank 6	0.003	403					

Collected on 23 Jan 01, analyzed on 25 & 27 Jan 01

GC #57244 Settings: Range=10, current=0.5, program: 40C-1min,15C/min-145C, hold 1min, 40C/min-220C, hold 1min

Tekmar 7000 Headspace Analyzer: 100uL (for highs) and 1mL (for lows) sample loops at 150C, transfer line at 150C flowing 12mL/min nitrogen, 50C for 50 min equil time, mix power 8 for 5 min

Column: J&W Science DB-VRX, 2.55 micron, length not recorded

Samples collected between 0900 and 1140 hrs

* Calibrations:

jan28low: AREA < 20354, TCE(ppb) = AREA/127559, r^2 = .9965, used zero, 0.1 and 1ppb standards (avgs)

jan28low: AREA > 20354, TCE (ppb) = 0.0000142464*AREA-0.340353, r^2 = 0.9998, Class VP generated using 6.85 to 536ppb standards

jan26hi: .00154233*AREA+8.76452

Table A.17 January 2001 tree-core TCE concentration data (high conc. samples)

Sample Type	x (ppb) *	y (area)	Head Space wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
hs-jan26hi samples							
modifier blank 1	ND	0					
modifier blank 2	ND	0					
6.85ppb	6.85	3988					
6.85ppb	6.85	4036					
6.85ppb	6.85	4587					
89.34ppb	89.34	58754					
89.34ppb	89.34	49330					
89.34ppb	89.34	61195					
268.02ppb	268.02	157198					
268.02ppb	268.02	146924					
268.02ppb	268.02	152768					
536.04ppb	536.04	343039					
536.04ppb	536.04	320548					
536.04ppb	536.04	380529					
804.06ppb	804.06	462051	not used for curve				
804.06ppb	804.06	379494	not used for curve				
804.06ppb	804.06	428100	not used for curve				
modifier blank 3	ND	0					
modifier blank 4	ND	0					
modifier blank 5	ND	0					
c3a	214.63	133474	2.058	9.500	0.761	2.146	2.82
c3b	197.78	122550	2.118	10.000	0.828	1.978	2.39
c3c	163.02	100014	2.012	9.500	0.764	1.630	2.13
c4a	145.22	88472	2.017	9.000	0.755	1.452	1.92
c4b	135.56	82212	2.275	10.500	0.858	1.356	1.58
c4c	147.25	89790	1.956	9.000	0.720	1.473	2.05
c10a	131.19	79378	1.863	9.000	0.820	1.312	1.60
c10b	128.06	77348	1.875	9.500	0.829	1.281	1.54
c10c	132.37	80143	1.936	10.000	0.894	1.324	1.48
modifier blank 6	ND	0					
modifier blank 7	ND	0					
cal chk #1 268.02ppb	291.82	183525					
modifier blank 8	ND	0					
modifier blank 9	ND	0					
c7a	41.82	21430	1.961	10.000	0.851	0.418	0.49
c7b	37.47	18614	2.059	10.000	0.896	0.375	0.42
c7c	36.44	17942	2.134	10.000	0.906	0.364	0.40
c11a	34.77	16860	2.026	10.000	0.841	0.348	0.41
c11b	37.86	18867	1.998	9.500	0.864	0.379	0.44
c11c	34.52	16701	2.030	9.500	0.853	0.345	0.40
modifier blank 10	ND	0					
cal chk #2 268.02ppb	265.58	166514					
modifier blank 11	ND	0					
modifier blank 12	ND	0					
trip blank 1	ND	0					
trip blank 2	ND	0					

Table A.18 February 2001 tree-core TCE concentration data
(low conc. samples)

Sample Type	Head Space						
	x (ppb) *	y (area)	wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
hs-feb15low samples							
0	0.00	0					
0.11	-0.34	15709					
0.11	-0.13	26989					
0.11	0.00	34029					
1.13	1.41	109194					
1.13	1.46	112064					
1.13	1.47	112549					
10.26	9.41	535433					
10.26	10.92	615903					
10.26	10.31	583576					
79.9		saturated					
79.9		saturated					
79.9		saturated					
119.84		saturated					
119.84		saturated					
119.84		saturated					
modifier blank #2		saturated					
modifier blank #3		saturated					
c1a		saturated	1.680	10.000	0.888	saturated	saturated
c1b		saturated	1.612	10.000	0.878	saturated	saturated
c1c		saturated	1.318	9.000	0.721	saturated	saturated
c2a	0.110	5221	2.074	10.000	1.108	0.001	< 0.00099
c2b	0.110	5187	2.123	10.000	1.118	0.001	< 0.00098
c2c	0.110	4024	1.969	10.000	1.044	0.001	< 0.0011
c8a	0.000	0	2.147	10.000	0.781	ND	ND
c8b	0.000	0	2.204	10.000	0.793	ND	ND
c8c	0.000	0	2.235	9.500	0.777	ND	ND
modifier blank #4	0.00	0					
calchk1 10.26ppb	12.57	703832					
modifier blank #5	0.00	0					

Collected on 15 Feb 01, analyzed on 19 Feb and 20 Feb 2001

GC#57244 Settings: Pgm: 100c-4min, 40c/min - 220c, hold 3min. Injection temp=200c
Column: DB-VRX 30m x 0.45mm, 2.55 um film thickness, nitrogen carrier gas at 12mL/min
Detector: Range=10, current=0.5, detector temp=300c, total flow = 50mL/min,
    purge flow = 2mL/min
Tekmar 7000: 50c-50min equilibration, mix power 8 for 5min,
    transfer line and sample loop temp=150c
Sample Loop volumes: 100ul (high samples), 1mL (low samples)
Samples collected between 0900 and 1100 hrs.

* Calibrations:
Feb15low: TCE (ppb) = 1.876e-005*AREA-0.638983 (linear regression from averages of all
feb15low standards), r^2=.9972
    tree c2 was reported as less than the lowest standard because area counts were < lowest area
    count from 0.11 standards
Feb15hi: TCE (ppb) = 0.00358895*AREA-0.299017
    (linear regression from averages of all feb15hi standards), r^2=0.9992

Table A.19 February 2001 tree-core TCE concentration data
(high conc. samples)

Sample Type	x (ppb) *	y (area)	wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
			Head Space				
hs-feb15hi samples							
modifier blank #1	0.00	0					
5.13ppb	5.13	1562					
5.13ppb	5.13	902					
5.13ppb	5.13	1495					
39.95ppb	39.95	12067					
39.95ppb	39.95	12536					
39.95ppb	39.95	10282					
199.74ppb	199.74	54276					
199.74ppb	199.74	62168					
199.74ppb	199.74	61219					
399.48ppb	399.48	107654					
399.48ppb	399.48	103025					
399.48ppb	399.48	103188					
998.7ppb	998.70	262277					
998.7ppb	998.70	280152					
998.7ppb	998.70	295830					
modifier blank #2		0					
modblk3		0					
c3a	69.46	19438	1.958	9.500	0.816	0.695	0.85
c3b	66.25	18543	1.932	10.000	0.759	0.663	0.87
c3c	65.63	18369	2.009	10.000	0.788	0.656	0.83
c4a	98.44	27511	1.999	8.500	0.702	0.984	1.40
c4b	74.75	20912	1.916	8.000	0.730	0.748	1.02
c10a	39.54	11100	1.936	9.500	0.898	0.395	0.44
c10b	46.67	13087	1.975	10.000	0.955	0.467	0.49
c10c	50.71	14212	2.027	9.500	0.926	0.507	0.55
modifier blank #4		0					
calchk1 199.74ppb	224.88	62742					
modifier blank #5		0					
c7a	15.07	4281	2.153	10.000	0.869	0.151	0.17
c7b	15.54	4414	2.110	10.000	0.849	0.155	0.18
c7c	13.06	3723	2.142	10.000	0.876	0.131	0.15
c11a	17.34	4916	2.172	9.500	0.877	0.173	0.20
c11b	18.11	5130	2.149	9.000	0.856	0.181	0.21
c11c	17.50	4960	2.228	9.500	0.891	0.175	0.20
modifier blank #6		0					
calchk2 199.74ppb	229.13	63926					
modifier blank #7		0					
trip blank1		0	-0.007				
trip blank2	1.19	414	-0.014				

Table A.20 March 2001 tree-core TCE concentration data
(low conc. samples)

Sample Type	Head Space						
	x (ppb) *	y (area)	wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
hs-mar13low samples							
0.029		43444					
0.15		74287					
3.71		337057					
37.14		1930079					
111.58		4043233					
148.77		6315997					
modifier blank #1		1193					
c1a	0.0290	16918	1.719	10.500	0.903	2.90E-04	0.000321
c1b	0.0290	11389	1.514	10.000	0.817	2.90E-04	0.000355
c1c	0.0290	11301	1.531	10.000	0.823	2.90E-04	0.000352
c2a	0.0290	3220	2.236	10.500	1.197	2.90E-04	0.000242
c2b	0.0290	2570	1.877	10.000	1.095	2.90E-04	0.000265
c2c	0.0290	2307	1.920	9.500	1.080	2.90E-04	0.000269
modifier blank #2		605					
c8a	ND	0	2.104	10.000	0.789	ND	ND
c8b	ND	0	2.012	9.500	0.809	ND	ND
c8c	ND	0	1.897	10.000	0.747	ND	ND
modifier blank #3		693					
0.029		51222					
0.15		59343					
3.71		336135					
37.14		1838487					
111.58		3596818					
148.77		5053655					

Collected on 13 Mar 01, analyzed on 15 & 21 Mar 01

GC#57244 Settings: Pgm: 60c-4min, 40c/min to 220c, hold 1min. Injection temp=200c
Column: DB-VRX NEW 30m x 0.45mm, 2.55 um film thickness, nitrogen carrier gas at 12mL/min
Detector: Range=10, current=0.05, detector temp=300c, total flow = 50mL/min,
    purge flow = 2mL/min
Tekmar 7000: 50c-50min equilibration, mix power 8 for 5min,
    transfer line and sample loop temp=180c
Sample Loop volumes: 100ul (high samples), 1mL (low samples)
Samples collected between 1230 and 1600 hrs.

* Calibrations:
mar13 low: no calibration used, samples were nearly 3 times lower than the lowest standard,
    reported as < .029ppb
mar13hi: ppb = (AREA - 1768.2)/254.33, used bracketed calibrations, r^2 = 0.9846
    (averages of 7.5 - 667ppb stds)

Table A.21 March 2001 tree-core TCE concentration data
(high conc. samples)

Sample Type	Head Space						
	x (ppb) *	y (area)	wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
hs-mar13hi samples							
2.83		631	not needed				
7.51	11.56	4707					
338.66	304.34	79172					
666.89	715.17	183657					
1163.75		256942	not needed				
1548.15		310569	not needed				
modifier blank #1	ND	0					
c3a	153.00	40680	1.686	9.000	0.668	1.530	2.29
c3b	170.27	45072	1.771	9.500	0.746	1.703	2.28
c3c	211.27	55501	2.090	9.500	0.831	2.113	2.54
modifier blank #3	ND	0					
c4a	101.34	27542	2.049	9.500	0.718	1.013	1.41
c4b	105.92	28706	2.031	9.500	0.733	1.059	1.44
c4c	129.58	34724	2.134	9.500	0.776	1.296	1.67
modifier blank #3	ND	0					
c10a	32.45	10021	1.910	10.000	0.919	0.324	0.35
c10b	33.88	10386	1.921	10.000	0.947	0.339	0.36
c10c	36.26	10991	1.854	10.000	0.946	0.363	0.38
modifier blank #4	ND	0					
c7a	15.64	5747	2.118	10.500	0.896	0.156	0.17
c7b	18.79	6548	2.067	10.500	0.897	0.188	0.21
c7c	25.57	8271	2.142	11.000	0.967	0.256	0.26
modifier blank #5	ND	0					
c11a	13.40	5176	2.321	10.500	0.942	0.134	0.14
c11b	10.29	4385	2.232	10.000	0.920	0.103	0.11
c11c	9.41	4161	2.214	10.000	0.936	0.094	0.10
modifier blank #6	ND	0					
u4-070 tree 1a	ND	0	1.849	9.000		ND	ND
u4-070 tree 1b	ND	0	1.763	9.000		ND	ND
u4-070 tree 2a	ND	0	2.272	10.500		ND	ND
u4-070 tree 2b	ND	0	1.932	10.500		ND	ND
modifier blank #7	ND	0					
2.83		385	not needed				
7.51	4.56	2927					
338.66	370.78	96068					
666.89	619.70	159377					
1163.75		246191	not needed				
1548.15		321555	not needed				

Notes: Well U4-070 is downgradient of well U4-065,
it sits north below the access road to well U4-065.

Table A.22 April 2001 tree-core TCE concentration data (low conc. samples)

Sample Type	Head Space						
	x (ppb) *	y (area)	wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
hs-apr3 low samples							
Avg of blanks	0.000	64					
0.000024		317	not used in calib curv				
0.000238		269	not used in calib curv				
0.00238	0.003	354					
0.02383	0.021	967					
0.23832	0.209	7080					
2.383	2.128	60301					
23.832		129674	not used in calib curv				
modifier blank #1		29					
c1a	0.029	1290	1.638	10.0	0.860	0.00029	0.00034
c1b	0.064	2162	1.794	10.5	0.903	0.00064	0.00071
c2a	0.050	1808	2.120	10.0	1.133	0.00050	0.00044
c2b	0.049	1796	2.058	9.5	1.135	0.00049	0.00043
c8a	-0.022	0	2.120	10.0	0.824	ND	ND
c8b	-0.022	0	2.062	9.5	0.798	ND	ND
modifier blank #2		99					
0.000024		326	not used in calib curv				
0.000238		300	not used in calib curv				
0.00238	0.003	374					
0.02383	0.020	929					
0.23832	0.208	7070					
2.383	2.152	60878					
23.832		618858	not used in calib curv				

Collected on 3 and 8 April 2001, analyzed 5 Apr (low) and 9 Apr (high samples)

GC#57244 Settings: Pgm: 60c-5min, 40c/min to 220c, hold 1min. Injection temp=200c
Column: DB-VRX NEW 30m x 0.45mm, 2.55 um film thickness, nitrogen carrier gas at 12mL/min
Detector: Range=10, current=1 (newly refurbished ECD#613523, temp=300c, total flow = 50mL/min,
    purge flow = 2mL/min
Tekmar 7000: 50c-50min equilibration, mix power 8 for 5min, transfer line and sample loop temp=180c
Sample Loop volumes: 100ul (high samples), 1mL (low samples)
Samples collected 1040-1330 hrs (4/3/01, rainy overcast) and 1500-1640 hrs
    (4/8/01, new snow on ground but sunny)

* calibrations:
apr5low: ppb = 9E-11*area^2 + 3E-5*area - 0.0081, r^2 = 1, range .00238 to 2.383ppb
    using avg of blanks as zero
apr8hi: ppb = 0.0164*area + 8.2423, r^2 = .9903, using 5.9 to 369ppb (bracketed calibration)

Notes: Sample descriptions contained N, W, NW etc.. Refer to which side of tree was sampled. Numbers after
the tree identification code (C-2,3,4, etc..) are height above ground in cm.

Table A.23 April 2001 tree-core TCE concentration data (high conc. samples)

Sample Type	x (ppb) *	y (area)	wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
hs-apr8hi samples			Head Space				
1.18	10.95	165	not used in calib curv				
5.91	12.88	283					
29.53	31.45	1415					
147.67	139.98	8033					
369.16	395.59	23619					
modifier blank #1		0					
c3a 76cm N agl	84.54	4652	1.661	8.5	0.627	0.845	1.35
c3b 76cm N agl	74.60	4046	1.463	8.5	0.605	0.746	1.23
c3a 242cm N agl	152.53	8798	2.054	10.0	0.758	1.525	2.01
c3b 242cm N agl	163.22	9450	2.044	10.0	0.757	1.632	2.16
modifier blank #2		0					
c3a 242cm S agl	271.95	16080	1.807	9.0	0.698	2.720	3.90
c3b 242cm S agl	269.07	15904	2.095	10.5	0.794	2.691	3.39
c3a 366cm E agl	243.83	14365	1.463	9.0	0.710	2.438	3.43
c3b 366cm E agl	309.79	18387	1.907	10.0	0.826	3.098	3.75
modifier blank #3		0					
c4a	183.85	10708	2.042	9.0	0.844	1.839	2.18
c4b	206.57	12093	2.042	8.5	0.848	2.066	2.44
c10a	159.01	9193	2.047	10.5	0.906	1.590	1.76
c10b	152.51	8797	1.852	10.0	0.859	1.525	1.78
modifier blank #4		0					
c7a	45.91	2297	2.189	10.5	0.899	0.459	0.51
c7b	47.54	2396	2.093	10.0	0.853	0.475	0.56
c11a	29.05	1269	1.937	9.0	0.774	0.291	0.38
c11b	31.45	1415	2.055	9.5	0.839	0.314	0.37
modifier blank #5		0					
field/trip blank1		0					
1.18		58	not used in calib curv				
5.91	12.90	284					
29.53	31.86	1440					
147.67	132.95	7604					
369.16	346.13	20603					

Table A.24 May 2001 tree-core TCE concentration data (low conc. samples)

Sample Type	Head Space						
	x (ppb) *	y (area)	wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
hs-may2 low samples							
0.021	0.019	671					
0.036	0.043	1273					
0.052	0.054	1542					
0.101	0.103	2737					
0.403	0.412	9993					
1.025	1.113	24909					
10.127		173963	not used				
modifier blank	ND	0					
c1a	0.146	3788	1.658	10.000	0.921	1.465E-03	0.001591
c1b	0.149	3853	1.736	10.000	0.937	1.492E-03	0.001593
c1c	0.155	3986	1.724	10.000	0.945	1.547E-03	0.001638
c2a	0.0112	476	1.979	10.000	1.112	1.119E-04	0.000101
c2b	0.0179	642	1.825	9.500	1.074	1.786E-04	0.00017
c2c	0.0209	718	1.850	9.500	1.148	2.092E-04	0.00018
c8a	ND	0	2.005	8.500	0.759	ND	ND
c8b	ND	0	1.903	8.500	0.725	ND	ND
c8c	ND	0	1.949	8.500	0.749	ND	ND
modifier blank	ND	0					
0.021	0.021	729					
0.036	0.041	1217					
0.052	0.060	1674					
0.101	0.127	3328					
0.403	0.464	11166					
1.025	1.205	26748	not used				

Collected on 2 May 2001, analyzed on 8 May (low), 9 May (hi)

GC#57244 Settings: Pgm: 60c-5min, 40c/min to 220c, hold 1min. Injection temp=200c
Column: DB-VRX NEW 30m x 0.45mm, 2.55 um film thickness, nitrogen carrier gas at 12mL/min
Detector: Range=10, current=.5 (refurbished ECD#613523, temp=300c, total flow = 50mL/min,
      purge flow = 2mL/min
Tekmar 7000: 50c-50min equilibration, mix power 8 for 5min, transfer line and sample loop temp=180c
Sample Loop volumes: 100ul (high samples), 1mL (low samples)
Samples collected 0950-1215 hrs, clear windy day

* Calibrations:
may9hi: ppb = 1E-07x2 + 0.0145x - 1.086, R2 = 0.9972, using 1.85 to 495 ppb
may8low: ppb = 2E-10x2 + 4E-05x - 0.0079, R2 = 0.9984, bracketed calibration of .0206 to 1.03ppb stds

Table A.25 May 2001 tree-core TCE concentration data (high conc. samples)

Sample Type	x (ppb) *	y (area)	wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
			Head Space				
hs-may2hi samples							
1.8536	1.7	193					
98.903	111.6	7110					
197.806	190.8	11535					
296.769	315.9	18525					
445.064	448.2	25917					
494.515	480.5	27725					
calchk 500	469.5	27107	Supelco 5000mg/L std, 1ul into 10ml modifier				
modifier blank	ND	0					
c3a	216.2	12953	1.603	10.000	0.740	2.162	2.92
c3b	230.5	13751	1.747	10.000	0.731	2.305	3.15
c3c	233.4	13917	1.541	9.000	0.680	2.334	3.43
c4a	192.9	11651	2.093	9.000	0.792	1.929	2.44
c4b	205.6	12359	2.108	9.000	0.796	2.056	2.58
c4c	202.9	12209	2.018	10.000	0.798	2.029	2.54
modifier blank	ND	0					
c10a	108.7	6945	2.004	9.500	0.842	1.087	1.29
c10b	132.9	8296	1.688	9.000	0.836	1.329	1.59
c10c	146.2	9044	1.939	10.000	0.865	1.462	1.69
c7a	15.0	1709	1.667	9.000	0.820	0.150	0.18
c7b	18.7	1916	1.678	9.000	0.837	0.187	0.22
c7c	26.1	2328	1.865	9.500	0.888	0.261	0.29
c11a	8.7	1356	1.877	9.000	0.879	0.087	0.10
c11b	14.4	1678	2.079	9.500	0.933	0.144	0.15
c11c	14.2	1667	2.082	9.500	0.899	0.142	0.16
modifier blank	ND	0					
c3a leaves	0.0	0	1.297	na		ND	ND
c3b leaves	0.0	0	1.773	na		ND	ND
c3c leaves	0.0	0	1.726	na		ND	ND
c3a buds	0.0	0	1.115	na		ND	ND
c3b buds	0.0	0	1.250	na		ND	ND
c3c buds	0.0	0	1.733	na		ND	ND

Table A.26 June 2001 tree-core TCE concentration data (low conc. samples)

Sample Type	x (ppb) *	y (area)	wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
hs-jun6 low samples							
Avg of blanks 1 & 2	0.000	296					
0.0242	0.0261	1394					
0.0242	0.0266	1409					
0.0242	0.0254	1371					
0.0242	0.0245	1340					
0.0242	0.0231	1293					
0.0242	0.0206	1212					
0.0242	0.0237	1313					
0.121	0.133	4896					
0.242	0.255	8784					
0.605	0.626	20064					
0.847	0.830	25942					
1.089	1.116	33890					
modifier blank #1	-0.007	299					
c1a	0.434	14308	1.380	9.5	0.791	0.00434	0.005484
c1b	0.056	2381	1.413	9.0	0.791	0.00056	0.000709
c1c	0.047	2072	1.339	9.0	0.767	0.00047	0.000609
c2a	0.190	6704	1.731	9.0	1.050	0.00190	0.001807
c2b	0.108	4090	1.253	9.0	0.685	0.00108	0.001583
c2c	0.121	4480	1.695	6.5	0.991	0.00121	0.001216
c8a	-0.007	298	1.919	9.0	0.732	< MDL	< MDL
c8b	-0.004	391	1.996	9.5	0.777	< MDL	< MDL
c8c	-0.005	364	2.281	11.0	0.923	< MDL	< MDL
modifier blank #2	-0.007	294					

Collected on 6 June 2001, analyzed on 8-9 June (low), 9-10 June (hi)

GC#57244 Settings: Pgm: 60c-5min, 40c/min to 220c, hold 1min. Injection temp=200c
Column: DB-VRX NEW 30m x 0.45mm, 2.55 um film thickness, nitrogen carrier gas at 12mL/min
Detector: Range=10, current=.5 (ECD#613523, temp=300c, total flow = 50mL/min, purge flow = 2mL/min
Tekmar 7000: 50c-50min equilibration, mix power 8 for 5min, transfer line and sample loop temp=180c
Sample Loop volumes: 100ul (high samples), 1mL (low samples)
Samples collected 1355-1645 hrs, clear day

* Calibrations: ......used new HP Chemstation software for data acquisition.
jun6low TCE(ppb) = 1E-10x2 + 3E-05x - 0.0159, R2 = 0.9991, used avg of blanks thru 1.089ppb stds
jun6hi: AREA > 6011 .. .0484*area - 23.928, r^2 = .9975, using 253 to 2500ppb stds
jun6hi: AREA < 6011 .. -1E-06*area^2 + .0485*area - 1.7557, r^2 = 0.9999, using .0242 to 253ppb stds
MDL calculated for 1mL & 100uL headspace sample loops using 99% confidence with 6 deg of freedom:
   Low (1mL loop): 0.006 ug/L in solution   High (100uL loop): 2.445 ug/L in solution

Notes: Sample descriptions contained N, W, NW etc.. Refer to which side of tree was sampled. Numbers after the tree identification code (C-2,3,4, etc..) are height above ground in cm.

Table A.27 8 June 2001 tree-core TCE concentration data (high conc. samples)

Sample Type	x (ppb) *	y (area)	wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
			Headspace				
hs-jun6hi samples							
1.962	3.42	107					
1.962	1.53	68					
1.962	1.83	74					
1.962	1.77	73					
1.962	1.31	63					
1.962	1.94	76					
1.962	0.98	57					
6.539	7.63	194					
101.358	101.28	2227					
253.4	253.63	6011					
405.4	434.05	9586					
506.8	501.76	10985					
1000	1040.26	22111					
1500	1477.64	31148					
2000	1900.98	39895					
2500	2547.63	53255					
modifier blank #1	ND	0					
c3-75a W	1964.82	41214	1.496	10.0	0.766	19.648	25.65
c3-75b W	2105.35	44117	1.513	9.5	0.719	21.053	29.28
c3-75c W	1894.30	39757	1.379	9.5	0.719	18.943	26.35
c3-240a W	1597.64	33628	1.708	9.5	0.736	15.976	21.71
c3-240b W	1631.67	34331	1.632	9.0	0.689	16.317	23.68
c3-240c W	1259.83	26648	1.630	9.0	0.707	12.598	17.82
c3-377a W	404.33	8972	1.451	9.0	0.669	4.043	6.04
c3-377b W	361.07	8079	1.419	8.0	0.686	3.611	5.26
c3-377c W	321.16	7254	1.357	8.5	0.686	3.212	4.68
modifier blank #2	ND	0					
cal chk 253.4ppb	276.43	6330					
c3-481a NW	277.47	6351	1.318	9.5	0.683	2.775	4.06
c3-481b NW	390.55	8688	1.382	9.0	0.667	3.905	5.86
c3-481c NW	276.27	6326	1.337	8.5	0.660	2.763	4.19
c3-617a N	656.13	14175	1.716	9.0	0.727	6.561	9.03
c3-617b N	996.42	21206	1.710	9.0	0.713	9.964	13.98
c3-617c N	1077.23	22875	1.661	8.5	0.714	10.772	15.09
c4a NW	815.80	17474	1.918	8.5	0.738	8.158	11.05
c4b NW	869.95	18593	1.691	7.5	0.632	8.700	13.77
c4c NW	939.82	20036	2.301	10.5	0.868	9.398	10.83
modifier blank #3	ND	0					
cal chk 253.4ppb	238.46	5599					
c10a W	148.00	3314	1.879	9.5	0.955	1.480	1.55
c10b W	134.70	2999	1.910	10.0	0.966	1.347	1.39
c10c W	162.46	3662	1.739	9.5	0.867	1.625	1.87
c7a S	92.83	2036	2.034	10.0	0.808	0.928	1.15
c7b S	101.88	2240	1.985	9.5	0.862	1.019	1.18
c7c S	89.31	1956	2.055	10.0	0.851	0.893	1.05
c11a E	92.82	2036	2.026	10.0	0.962	0.928	0.96
c11b E	91.44	2004	2.134	11.0	1.007	0.914	0.91
c11c E	82.57	1806	2.120	11.0	0.998	0.826	0.83
trip blank	ND	0					
cal chk 500ppb	498.32	10914	used 1ul of supelco 5000ppm std				
cal chk 500ppb	484.63	10631	used 1ul of supelco 5000ppm std				

June 6 High Calibration (253 to 2500ppb)

$y = 0.0484x - 23.928$

$R^2 = 0.9975$

TCE (ug/L) vs AREA
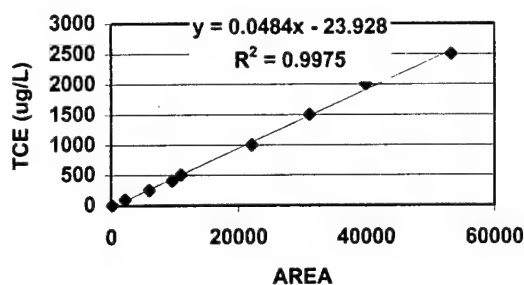
Table A.28 28 June 2001 tree-core TCE concentration data (high conc. samples)

Sample Type	Headspace						
	x (ppb) *	y (area)	wet core wt (g)	core length (cm)	dry core wt (g)	mass TCE (ug)	ug/g (TCE in dry core)
hs-jun28 high samples							
0.927	-10.291	509					
92.53	97.808	7479					
462.65	458.721	30749					
925.3	938.735	61698					
1850.6	1906.462	124093					
2775.9	2734.667	177492					
field blank 1	ND	0					
field blank 2	ND	0					
c3a (1431hrs) NE	342.650	23265	1.183	8.5	0.626	3.427E+00	5.48
c3b (1441hrs) NE	146.710	10632	1.666	9.0	0.661	1.467E+00	2.22
c3c (1451hrs) NE	202.970	14259	1.798	9.0	0.726	2.030E+00	2.80
c4a (1320hrs) SE	139.4100	10161	1.740	7.5	0.626	1.394E+00	2.23
c4b (1400hrs) SE	147.2400	10666	1.653	7.5	0.624	1.472E+00	2.36
c4c (1408hrs) SE	160.8100	11541	1.473	6.5	0.544	1.608E+00	2.96
c10a (1540hrs) SW	76.410	6099	1.602	10.0	0.793	7.641E-01	0.96
c10b (1550hrs) SW	79.210	6280	1.462	9.0	0.781	7.921E-01	1.01
c10c (1555hrs) SW	80.340	6353	1.703	9.5	0.818	8.034E-01	0.98

Collected on 28 June 2001, analyzed on 3 July 01

GC #57244 Settings: 60c-5min, 40c/min to 220, hold 1min, injection temp = 200C
Column: J&W Science Cat no 1241574, DB-VRX, 2.55 micron, 30m x 0.45mm
Detector settings: Range=10, current = .5 , temp = 300C
Tekmar 7000: 50c-50min equilibration, mix power 8 for 5min, transfer line and sample loop temp=180c
Tekmar Sample Loop volume: 100ul
Samples collected 1300-1555 hrs, clear day. Sample description include time of day and radial side of tree
    where sample was collected.

* Calibration:
TCE (ppb) = (AREA -1172.69366)/64.47561 , r^2 = 0.9996, used all standards

Notes: Used new HP data acquisition software for this data set.

Appendix B.

Purge and Trap Analyses Data for April 2001

Table B.1 April 2001 analysis of tree cores using purge and trap with GC/MS.

Sample or Standard	Purge & Trap		core wt (wet)	core wt (dry) calculated	Volume MeOH in sample, VMeOH(mL)	Volume of DDW First Dilution, VDDW (mL)	Aliquot Volume of Meoh Injected into PT Water (mL)	Dilution Factor (VDDW/aliquot volume)	Conc in MeOH (ppb) [ppb][Dilution Factor] volume	mass TCE (mg) [Conc in MeOH] VMeOH	MeOH mg/kg (TCE in dry core)
	y (ppb)*	x (area)									
0.01454	0.0117	335.00									
0.07259	0.0775	833.00									
0.14519	0.1418	1357.00									
0.43556	0.4352	4688.00									
c1 SW	0.0089	314.00	1.390	0.675	39.478	40.784	500	81.569	0.723	0.029	0.0422
c1 SW	0.0038	277.00	1.579	0.767	39.535	40.660	500	81.319	0.308	0.012	0.0158
c2 SW	-0.0350	0.00	1.680	0.768	39.671	40.573	500	81.147	-2.840	-0.113	BDL
c2 SW	-0.0350	0.00	1.833	0.838	39.733	40.514	500	81.029	-2.836	-0.113	BDL
c8 N	0.0071	301.00	1.990	1.218	38.886	41.485	500	82.971	0.587	0.023	0.0187
c8 N	-0.0350	0.00	1.837	1.125	38.532	42.218	500	84.436	-2.955	-0.114	BDL
c3 N 76agl	0.1121	1110.00	1.791	1.083	37.119	40.646	25	1625.828	182.233	6.764	6.2478
c3 N 76agl	0.0914	943.00	1.787	1.080	38.335	40.713	25	1628.520	148.805	5.704	5.2807
c3 N 242agl	0.0970	988.00	1.957	1.221	38.913	41.000	25	1639.988	159.087	6.190	5.0710
c3 N 242agl	0.1226	1196.00	1.863	1.162	38.503	41.529	25	1661.140	203.583	7.839	6.7450
c3 S 242agl	0.2430	2279.00	1.800	1.123	37.367	40.315	25	1612.584	391.879	14.643	13.0412
c3 S 242agl	0.2697	2548.00	1.746	1.089	36.820	41.196	25	1647.856	444.360	16.362	15.0222
c3 E 366agl	0.2432	2281.00	1.933	1.045	38.714	42.071	25	1682.820	409.288	15.845	15.1577
c3 E 366agl	0.2670	2521.00	1.653	0.894	36.685	41.376	25	1655.028	441.965	16.214	18.1371
c4 NE	0.1188	1165.00	2.137	1.252	39.737	41.303	25	1652.136	196.270	7.799	6.2312
ptwblank	-0.0350	0.00									
c4 NE	0.1360	1308.00	2.138	1.252	38.922	40.514	25	1620.568	220.385	8.578	6.8500
c10 SW	0.1489	1417.00	1.903	1.041	39.458	39.883	25	1595.312	237.465	9.370	9.0046
c10 SW	0.1449	1383.00	1.879	1.027	38.401	41.287	25	1651.484	239.239	9.187	8.9416
c7 SW	0.0636	725.00	1.895	1.120	38.542	41.349	50	826.970	52.580	2.027	1.8098
c7 SW	0.0848	891.00	1.952	1.153	38.517	40.969	50	819.370	69.501	2.677	2.3209
c11 S	0.0385	534.00	1.822	1.086	39.534	41.713	50	834.252	32.143	1.271	1.1700
c11 S	0.0379	529.00	1.924	1.147	38.378	41.037	50	820.736	31.076	1.193	1.0399
origPTW blank	-0.0350	0.00									

Collected on 3(low) and 8(med/hi) Apr 01, analyzed on 22Apr01

P&T analysis using gc-ms selective ion monitoring, tce retention time ~6.8
PT method: pkerry.m .... 5ml purge volume, 11min purge time, 2.5min purge
GCMS method: ptcesim.m for samples, ptcesma.m for 3rd run stds, Chemstation integrator, integration parameters kerry.e
GCMS sequence:

Samples collected 1040-1330 hrs (4/3/01, rainy overcast) and 1500-1640 hrs (4/8/01, new snow on ground but sunny)

Dry weights calculated using dry weight fractions for each tree taken from April 2001 GC-ECD results.

* Calibration:
ppb (or ug/L) = -.035 + 1.425E-4*AREA - 9E-9*AREA^2, r^2=1, using Stat View

Table B.2 April 2001 analysis of tree cores using purge and trap with GC/ECD.

Sample or Standard	x (ppb) *	y (area)	core wt (wet)	core wt (dry)	Volume MeOH in sample, VMeOH(mL)	Volume of DDW First Dilution, VDDW (mL)	Aliquot Volume of Meoh Injected into PT Water (mL)	Dilution Factor [VDDW]/aliquot volume	Conc in MeOH (ppb) [ppb][Dil Factor]	mass TCE (mg) [Conc in MeOH]* VMeOH	PT-GC/ECD mg/kg (TCE in dry core)	Headspace GC/ECD mg/kg (TCE in dry core)	PT-GC/MS mg/kg (TCE in dry core)
0.00126	-0.0074	73797											0.042
0.0125	0.0137	130084											0.016
0.0616	0.0667	271209											BDL
0.1232	0.1281	434713											BDL
0.4312	0.4288	1235647											0.019
origPTW blank	-0.0271	21448											BDL
newPTWorig250ulmeoh	-0.0281	18861											
c1 SW	-0.0178	46303	1.390	0.675	39.478	41.727	250	166.91	-2.965	-0.117	BDL	0.000339	0.042
c1 SW	-0.0030	85681	1.579	0.767	39.535	42.466	250	169.86	-0.506	-0.020	BDL	0.000706	0.016
c2 SW	-0.0143	55606	1.680	0.768	39.671	42.001	250	168.00	-2.397	-0.095	BDL	0.000439	BDL
c2 SW	-0.0099	67318	1.833	0.838	39.733	41.634	250	166.54	-1.644	-0.065	BDL	0.000434	BDL
c8 N	-0.0163	50140	1.990	1.218	38.886	41.346	250	165.38	-2.699	-0.105	BDL	ND	0.019
c8 N	-0.0189	43309	1.837	1.125	38.532	42.460	250	169.84	-3.208	-0.124	BDL	ND	BDL
c3 N 76agl	0.2743	824349	1.791	1.083	37.119	41.446	25	1657.84	454.828	16.883	15.59	1.35	6.25
c3 N 76agl	0.0615	257442	1.787	1.080	38.335	42.514	25	1700.56	104.598	4.010	3.71	1.23	5.28
c3 N 242agl	0.0633	262124	1.957	1.221	38.913	41.680	25	1667.20	105.476	4.104	3.36	2.01	5.07
c3 N 242agl	0.0878	327557	1.863	1.162	38.503	41.590	25	1663.60	146.117	5.626	4.84	2.16	6.74
c3 S 242agl	0.1859	588821	1.800	1.123	37.367	41.607	25	1664.28	309.426	11.562	10.30	3.90	13.04
c3 S 242agl	0.2068	644389	1.746	1.089	36.820	41.415	25	1656.60	342.560	12.613	11.58	3.39	15.02
c3 E 366agl	0.2024	632646	1.933	1.045	38.714	41.729	25	1669.16	337.798	13.078	12.51	3.43	15.16
c3 E 366agl	0.2203	680391	1.653	0.894	36.685	41.151	25	1646.04	362.625	13.303	14.88	3.75	18.14
c4 NE	0.0893	331425	2.137	1.252	39.737	41.842	25	1673.68	149.433	5.938	4.74	2.18	6.23
c4 NE	0.0926	340382	2.138	1.252	38.922	41.103	25	1644.12	152.323	5.929	4.73	2.44	6.85
c10 SW	0.1050	373290	1.903	1.041	39.458	41.960	25	1678.40	176.236	6.954	6.68	1.76	9.00
c10 SW	0.1039	370331	1.879	1.027	38.401	41.667	25	1666.68	173.153	6.649	6.47	1.78	8.94
c7 SW	0.0355	188172	1.895	1.120	38.542	43.259	50	865.18	30.715	1.184	1.06	0.51	1.81
c7 SW	0.0726	286916	1.952	1.153	38.517	41.563	50	831.26	60.328	2.324	2.01	0.56	2.32
c11 S	0.0186	143080	1.822	1.086	39.534	42.000	50	840.00	15.600	0.617	0.57	0.38	1.17
c11 S	0.0175	140328	1.924	1.147	38.378	42.000	50	840.00	14.732	0.565	0.49	0.37	1.04
origPTW blank	-0.0287	17043											

Collected on 3(low) and 8(med/hi) Apr 01, analyzed on 22Apr0 for purge and trap analyis:

GC #57244 Settings: program: 60C-5min, 40C/min to 220C, hold 1 min (10min program), inject temp = 200C, nitrogen purge/carrier gas, carrier flow = 12mL/min

ECD settings: Range=10, current=0.5, temp = 300C, nitrogen total flow = ~50mL/min

P&T Settings: Prepurge=0min, S. Preheat temp=40C,W. Preheat temp=40C, stir disabled, preht time=0, purge time=11m, dry purge=2min, desorb preht=50C

P&T Settings: desorb temp=180C, desorb time=4m, bake temp=220C, bake time=7min, trsfr line=170C, valve oven=150C,gc cycle time=0min. Flushes=3, sample volume=10ml

Column: J&W Science, DB-VRX, 2.55 micron, 30m x 0.45mm

Samples collected 1040-1330 hrs (4/3/01, rainy overcast) and 1500-1640 hrs (4/8/01, new snow on ground but sunny)

* Calibration:

linear formula for P&T: ppb = 3.75444e-7*AREA-0.0351473, r^2=0.9989, cal range (.00126-0.4312ppb)

Appendix C.

2001 OU-4 Well Water Data

Table C. Hill AFB site OU-4 2001 well water data

Site	Well	19-Jan-01		13-Feb-01		6-Mar-01		3-Apr-01		2-May-01		6-Jun-01	
		TCE Conc. (µg/L)	Depth to Groundwater (feet)	TCE Conc. (µg/L)	Depth to Groundwater (feet)	TCE Conc. (µg/L)	Depth to Groundwater (feet)	TCE Conc. (µg/L)	Depth to Groundwater (feet)	TCE Conc. (µg/L)	Depth to Groundwater (feet)	TCE Conc. (µg/L)	Depth to Groundwater (feet)
Control	u4-037			0.1 U 0.2 F	17.49	0.1 U	16.21	0.6 F	16.11			0.10	16.02
C	u4-075	0.2 F		0.3 F	15.91	0.1 U	15.62	0.1 U	15.58	0.1 U	15.48	0.1 U	15.61
	u4-076	0.2 U		0.4 F	15.31	0.1 U	14.92	0.4 F	14.68	0.4 F	14.43	0.1 F	14.79
B	u4-062	1020	21.02	1380	20.76	1270	20.62	1570	20.45	1150		1300 1350	20.28
	u4-064	669 719	17.48	886	17.30	743	17.12	1660	16.73	754 746	16.12	832	16.98
A	u4-006	4610	16.41	4890	15.58	4190 4560	15.15	4620	15.08	3720	14.52	4640	16.50

Notes:
Blank cells = No reading taken
F = TCE positively identified, but below the reporting limit of 5 µg/L
U = TCE not detected, value is at or below the MDL of 0.1 µg/L

Appendix D.

Stable Isotope Data for OU-4 Sourcewater Evaluation

Table D. Experimentally derived hydrogen isotope ratios for tree
tissue, groundwater, and rainwater at Hill AFB site OU-4.

Sample	Type	Isotope Ratio	Plot Location	Date Collected	Date Analyzed
c3	Cottonwood	-122	1	10/16/96	5/23/97
c3	Cottonwood	-124	1	10/16/96	5/23/97
c1	Box Elder	-106	5	3/12/97	5/23/97
c2	Apple	-102	5	3/12/97	5/23/97
c3	Cottonwood	-117	5	3/12/97	5/23/97
c4	Russian Olive	-123	5	3/12/97	5/23/97
c7	Poplar	-119	5	3/12/97	5/23/97
c8	Russian Olive	-119	5	3/12/97	5/23/97
c10	Poplar	-109	5	3/12/97	5/23/97
c11	Poplar	-114	5	3/12/97	5/23/97
c1	Box Elder	-110	6	4/2/97	5/23/97
c2	Apple	-106	6	4/2/97	5/23/97
c3	Cottonwood	-110	6	4/2/97	5/23/97
c4	Russian Olive	-127	6	4/2/97	5/23/97
c7	Poplar	-119	6	4/2/97	5/23/97
c8	Russian Olive	-125	6	4/2/97	5/23/97
c10	Poplar	-123	6	4/2/97	5/23/97
c11	Poplar	-118	6	4/2/97	5/23/97
U4-006	Groundwater	-120	2	10/16/96	5/23/97
U4-065	Groundwater	-123	2	10/16/96	5/23/97
c1	Box Elder	-129	7	6/5/97	7/17/97
c2	Apple	-123	7	6/5/97	7/17/97
c3	Cottonwood	-126	7	6/5/97	7/17/97
c4	Russian Olive	-142	7	6/5/97	7/17/97
c7	Poplar	-150	7	6/5/97	7/17/97
c8	Russian Olive	-128	7	6/5/97	7/17/97
c10	Poplar	-131	7	6/5/97	7/17/97
c11	Poplar	-136	7	6/5/97	7/17/97
U4-075	Groundwater	-122	3	6/5/97	7/17/97
U4-064	Groundwater	-122	3	6/5/97	7/17/97
U4-062	Groundwater	-119	3	6/5/97	7/17/97
U4-006	Groundwater	-118	3	6/5/97	7/17/97
U4-076	Groundwater	-124	3	6/5/97	7/17/97
U4-037	Groundwater	-122	3	6/5/97	7/17/97
rainwater	Surfacewater	-149	4	6/11/97	7/17/97

Plot Location only used to arrange the results on the chart.
All ratios reported using SMOW, standard mean ocean water.

Appendix E.

Temperature and Precipitation Data

Table E. Daily surface weather data provided by the National Climatic Data Center (via internet) for Ogden Pioneer P H and Ogden Sugar Factory weather stations.

	24-Jan	28-Feb	9-Mar	13-Apr	18-May	8-Jun	31-Jul	31-Aug	2-Oct	2-Nov	23-Jan	15-Feb	13-Mar	3,8-Apr	2-May	6-Jun	28-Jun
Max Temp (degF)	35	54	48	75	68	97	99	70	81	43	35	29	46	57	62	73	91
SNOW (tenths of inch)	0	0	30	0	0	0	0	0	0	0	0	3	0	0	0	0	0
Precipitation (hundredths of inch)	0	27	45	0	3	0	0	27	0	0	0	na	0	50	0	0	0

May 2000 - unavailable from Ogden Pioneer P H weather station...used Ogden Sugar Factory instead. For 3 and 8 Apr data an average of the two was used. (Precip 3 Apr = 0, 8 Apr = 99) (Temp 3 Apr = 53, 8 Apr = 70)

Appendix F.

Headspace Optimization Test Data

Table F. Optimization test run data for the Tekmar 7000
headspace analyzer on 5-7 Jan 2000

25c	20min		40min		60min		120min	
TCE	201306	198203	291017	300474	308491	312168	303864	303442
TCA	24413	24228	25581	25962	25221	25223	24853	24410
PCE	672226	632225	1048782	1095849	1163297	1184003	1141438	1171933

35c	20min		40min		60min		120min	
TCE	240278	278872	307195	305490	314125	319246	319762	311950
TCA	28897	29068	29884	29235	30821	29934	30150	30160
PCE	815391	977928	1132970	1139014	1190499	1215595	1210028	1192717

45c	20min		40min		60min		120min	
TCE	269375	256331	315750	320355	326194	321300	321280	332890
TCA	27790	26010	34155	29671	37592	34280	37444	38644
PCE	953599	867751	1183648	1221974	1241846	1240484	1212708	1259114

55c	20min		40min		60min		120min	
TCE	277093	241190	321036	319289	332493	201527	309847	336405
TCA	30853	32598	34336	34026	44000	30895	44085	45481
PCE	1013001	758870	1229984	1226493	1287670	493083	1161722	1290739

65c	20min		40min		60min		120min	
TCE	273829	292040	329387	318797	323173	319839	333130	329506
TCA	38656	39188	44972	39030	47570	41421	52467	51151
PCE	971880	1122430	1306093	1262505	1303227	1294223	1307994	1303890

TCE - trichloroethylene, TCA - trichloroethane, PCE - perchloroethene
(values listed are area counts from Shimadzu Class VP data acquisition printed reports)

Appendix G.

Wood Sorption/Desorption Data

Table G.1. Sorption kinetics study data used to determine equilibration time for sorption experiment.

Time from spike (hrs)	0		3				6.75			
Sample	dpm/mL	WaterConc /Initial Conc (normalized)	dpm	Volume of Sample Injected (ul)	Conc. (dpm/mL)	WaterConc /Initial Conc (normalized)	dpm	Volume of Sample Injected (ul)	Conc. (dpm/mL)	WaterConc /Initial Conc (normalized)
Cottonwood	101744.59	1.00	357.76	5	71552.0	0.703	315.75	5	63150.0	0.621
Cottonwood	101455.83	1.00	338.15	5	67630.0	0.667	328.15	5	65630.0	0.647
Cottonwood	99934.25	1.00	344.31	5	68862.0	0.689	320.94	5	64188.0	0.642
Average	101044.89	1.00			69348.0	0.686			64322.67	0.637
Std. Dev.	972.61	0.00			2005.66	0.018			1245.47	0.014
Russian Olive	100691.35	1.00	369.94	5	73988.0	0.735	368.48	5	73696.0	0.732
Russian Olive	100464.35	1.00	364.1	5	72820.0	0.725	357.56	5	71512.0	0.712
Russian Olive	102311.12	1.00	344.33	5	68866.0	0.673	353.54	5	70708.0	0.691
Average	101155.61	1.00			71891.33	0.711			71972.00	0.712
Std. Dev.	1007.12	0.00			2684.31	0.033			1546.20	0.020
Control 1	95213.69	1.00	499.96	5	99992.0	1.050	487.68	5	97536.0	1.024
Control 2	95866.16	1.00	488.19	5	97638.0	1.018	486.98	5	97396.0	1.016
Control 3	96112.22	1.00	498.54	5	99708.0	1.037	507.76	5	101552.0	1.057
Average	95539.93	1.00			99112.67	1.035			98828.00	1.032
Std.Dev.	461.37	0.00			1284.97	0.016			2360.09	0.021

Table G.1. (cont)

Time from spike (hrs)		12.75				24.25				48.5			
Sample	dpm	Volume of Sample Injected (ul)	Conc. (dpm/mL)	WaterConc /Initial Conc (normalized)	dpm	Volume of Sample Injected (ul)	Conc. (dpm/mL)	WaterConc /Initial Conc (normalized)	dpm	Volume of Sample Injected (ul)	Conc. (dpm/mL)	WaterConc /Initial Conc (normalized)
Cottonwood	632.03	10	63203.0	0.621	605.84	10	60584.0	0.595	570.65	10	57065.0	0.561
Cottonwood	618.67	10	61867.0	0.610	598.55	10	59855.0	0.590	562.56	10	56256.0	0.554
Cottonwood	645.87	10	64587.0	0.646	609.64	10	60964.0	0.610	592.19	10	59219.0	0.593
Average			63219.00	0.626			60467.67	0.598			57513.33	0.569
Std. Dev.			1360.07	0.019			563.58	0.010			1531.53	0.020
Russian Olive	675.28	10	67528.0	0.671	648.48	10	64848.0	0.644	637.34	10	63734.0	0.633
Russian Olive	682.11	10	68211.0	0.679	676.84	10	67684.0	0.674	657.9	10	65790.0	0.655
Russian Olive	667.25	10	66725.0	0.652	659.85	10	65985.0	0.645	630.69	10	63069.0	0.616
Average			67488.00	0.667			66172.33	0.654			64197.67	0.635
Std. Dev.			743.81	0.014			1427.25	0.017			1418.52	0.019
Control 1	931.76	10	93176.0	0.979	931.88	10	93188.0	0.979	865.41	10	86541.0	0.909
Control 2	939.93	10	93993.0	0.980	922.65	10	92265.0	0.962	859.99	10	85999.0	0.897
Control 3	955.97	10	95597.0	0.995	924.84	10	92484.0	0.962	907.66	10	90766.0	0.944
Average			94255.33	0.985			92645.67	0.968			87768.67	0.917
Std.Dev.			1231.63	0.009			482.27	0.009			2609.87	0.025

Table G.1. (cont)

Time from spike (hrs)			72.5		
Sample	dpm	Volume of Sample Injected (ul)	Conc. (dpm/mL)		WaterConc /Initial Conc (normalized)
Cottonwood	552.75	10	55275.0		0.543
Cottonwood	552.49	10	55249.0		0.545
Cottonwood	561.96	10	56196.0		0.562
Average			55573.33		0.550
Std. Dev.			539.40		0.011
Russian Olive	613.63	10	61363.0		0.609
Russian Olive	638.26	10	63826.0		0.635
Russian Olive	619.31	10	61931.0		0.605
Average			62373.33		0.617
Std. Dev.			1289.70		0.016
Control 1	827	10	82700.0		0.869
Control 2	843.26	10	84326.0		0.880
Control 3	883.26	10	88326.0		0.919
Average			85117.33		0.889
Std.Dev.			2895.28		0.027

Table G.2   Sorption/desorption data

Vial #	Vial Wt. (g)	With Tissue (g)	Tissue Wt (g)	W/Water Wt (g)	Water Vol (mL)	W/Cap Wt (g)	Cap Weight (g)	Vial/tissue /cap weight (g)
DW21	15.970	na	na	36.70	20.73	38.19	1.49	17.46
DW20	15.798	na	na	36.34	20.54	37.84	1.50	17.30
DW19	15.762	na	na	36.36	20.60	38.00	1.64	17.40
DW18	15.661	na	na	35.78	20.11	37.39	1.61	17.28
DW17	15.735	na	na	36.16	20.42	37.75	1.60	17.33
DW16	15.701	na	na	36.27	20.57	37.91	1.64	17.34
DW15	15.688	na	na	36.35	20.66	37.90	1.55	17.24
DW14	15.703	na	na	36.28	20.58	37.91	1.63	17.33
DW13	15.659	na	na	35.86	20.20	37.51	1.65	17.31
DW12	15.632	na	na	36.02	20.39	37.65	1.63	17.26
DW11	15.859	na	na	36.48	20.62	38.11	1.63	17.49
DW10	15.860	na	na	36.48	20.62	38.11	1.62	17.48
DW9	15.723	na	na	35.91	20.19	37.54	1.63	17.36
DW8	15.736	na	na	36.16	20.42	37.79	1.63	17.37
DW7	15.895	na	na	36.33	20.43	37.93	1.60	17.50
DW6	15.791	na	na	36.04	20.25	37.68	1.64	17.43
DW5	15.893	na	na	36.28	20.39	37.92	1.64	17.53
DW4	15.753	na	na	35.98	20.23	37.59	1.61	17.36
DW3	15.808	na	na	36.43	20.62	38.08	1.65	17.45
DW2	15.814	na	na	36.31	20.49	37.95	1.65	17.46
DW1	15.762	na	na	36.49	20.73	38.08	1.59	17.35

Table G.2

Vial #	Spiked Counts (dpm/mL)	Spiked Counts (total dpm)	24hr Count (dpm)	24 Obs Counts (dpm/ml)	24 Obs Counts (total dpm)	24 Est Tiss Count (dpm/g)	W/O Water (g)	W/ Water (g)
DW21	15654.72	324569.21	29439.20	14699.85	304771.91	na	33.19	38.05
DW20	15803.35	324569.21	28019.85	13990.17	287330.14	na	32.86	37.74
DW19	15757.32	324569.21	28544.62	14252.56	293574.16	na	33.00	37.87
DW18	3269.72	65767.11	6337.38	3148.94	63337.71	na	32.27	37.17
DW17	3220.56	65767.11	6452.18	3206.34	65476.59	na	32.67	37.54
DW16	3197.55	65767.11	6406.02	3183.26	65473.22	na	32.85	37.79
DW15	1583.08	32708.06	3431.12	1695.81	35037.05	na	32.77	37.59
DW14	1589.39	32708.06	3516.01	1738.25	35771.47	na	32.92	37.78
DW13	1618.89	32708.06	4225.23	2092.86	42284.17	na	32.51	37.40
DW12	90393.34	1843210.63	181333.97	90647.23	1848387.69	na	32.47	37.39
DW11	89385.12	1843210.63	142845.13	71402.81	1472397.37	na	33.08	37.96
DW10	89380.79	1843210.63	177931.91	88946.20	1834248.56	na	33.15	38.00
DW9	40831.53	824306.87	87450.53	43705.51	882326.86	na	32.44	37.28
DW8	40367.62	824306.87	82991.25	41475.87	846937.29	na	32.75	37.64
DW7	40347.86	824306.87	81641.27	40800.88	833562.01	na	32.89	37.76
DW6	8474.20	171627.92	19327.77	9644.13	195322.59	na	32.66	37.73
DW5	8417.26	171627.92	17727.26	8843.88	180326.64	na	33.01	37.91
DW4	8483.41	171627.92	16919.87	8440.18	170753.31	na	32.55	37.35
DW3	0.00	0.00	39.87	BDL	BDL	na	33.09	38.01
DW2	0.00	0.00	36.16	BDL	BDL	na	32.91	37.73
DW1	0.00	0.00	45.84	3.17	65.64	na	33.06	37.94

Table G.2

Vial #	Mass Removed (dpm)	Expect Mass (calc) (dpm)	48hr Count (dpm)	48 Obs Counts (dpm/ml)	48 Obs Counts (total dpm)	48 Est Tiss Count (dpm/g)	W/O Water (g)	W/ Water (g)	Mass Removed (dpm)
DW21	73499.23	231272.68	20418.65	10189.57	209833.84	na	33.09	37.98	50540.27
DW20	69629.08	217701.06	19873.55	9917.02	202713.83	na	32.78	37.62	49188.43
DW19	71277.03	222297.12	20048.90	10004.70	204766.12	na	32.33	37.16	55426.02
DW18	16119.41	47218.30	3124.32	1542.41	30686.17	na	32.15	37.01	7742.88
DW17	16301.01	49175.58	4116.21	2038.35	41188.97	na	32.51	37.46	10252.91
DW16	16094.54	49378.67	4276.57	2118.53	43328.20	na	32.74	37.67	10698.58
DW15	8692.70	26344.35	2053.44	1006.97	20496.80	na	32.62	37.47	5004.62
DW14	8670.40	27101.08	1774.27	867.38	17738.82	na	32.78	37.69	4336.91
DW13	10462.21	31821.96	2310.85	1135.67	22821.32	na	32.35	37.22	5735.14
DW12	469462.01	1378925.68	95881.78	47921.14	964748.32	na	32.42	37.35	238168.05
DW11	359084.74	1113312.63	76401.26	38180.88	781638.90	na	32.99	37.92	189758.96
DW10	440817.37	1393431.19	99635.27	49797.88	1021653.33	na	33.02	37.94	247993.45
DW9	223072.93	659253.93	42405.02	21182.76	422045.24	na	32.24	37.22	106761.09
DW8	208872.49	638064.80	48351.30	24155.90	489736.64	na	32.60	37.52	121745.72
DW7	205514.04	628047.97	55215.37	27587.93	559014.20	na	32.74	37.69	138491.40
DW6	48432.83	146889.76	9838.34	4899.42	99463.05	na	32.74	37.66	24448.09
DW5	43449.96	136876.67	8887.51	4424.00	90147.88	na	32.94	37.88	21987.29
DW4	42572.27	128181.03	11309.63	5635.06	112627.97	na	33.39	37.27	22314.84
DW3	BDL	BDL	47.44	3.97	81.54	na	33.04	37.96	19.71
DW2	BDL	BDL	60.16	10.33	209.31	na	32.76	37.63	51.32
DW1	15.89	49.75	56.88	8.69	178.87	na	32.91	37.80	43.69

Table G.2

Vial #	Expect Mass (calc) (dpm)	72hr Count (dpm)	72 Obs Counts (dpm/ml)	72 Obs Counts (total dpm)	72 Est Tiss Count (dpm/g)	W/O Water (g)	W/ Water (g)	Mass Removed (dpm)
DW21	159293.57	15253.10	7606.80	156114.28	na	32.99	38.08	37957.91
DW20	153525.41	15709.20	7834.85	159211.91	na	32.65	37.53	38939.19
DW19	149340.10	11151.82	7408.21	146364.02	na	32.19	37.26	36818.81
DW18	22943.29	1807.85	884.17	17449.12	na	32.04	37.00	4394.33
DW17	30936.06	2058.39	1009.44	20317.03	na	32.49	37.55	5016.92
DW16	32629.62	2121.50	1041.00	21165.54	na	32.66	37.62	5215.39
DW15	15492.18	1184.94	572.72	11588.92	na	32.48	37.51	2857.85
DW14	13401.91	978.26	469.38	9556.97	na	32.66	37.58	2360.96
DW13	17086.18	1170.07	565.28	11257.58	na	32.22	37.24	2826.41
DW12	726580.27	50546.88	25253.69	507397.07	na	32.38	37.32	125510.82
DW11	591879.94	40446.76	20203.63	412800.49	na	32.90	37.91	101422.20
DW10	773659.88	44125.98	22043.24	450916.44	na	32.95	37.88	109995.75
DW9	315284.15	25368.61	12664.55	251568.65	na	32.25	37.28	62942.82
DW8	367990.92	31329.13	15644.81	315305.53	na	32.46	37.35	79162.75
DW7	420522.80	31384.39	15672.44	316473.56	na	32.65	37.66	78989.09
DW6	75014.96	5641.38	2800.94	56665.74	na	32.45	37.39	14592.88
DW5	68160.59	4560.03	2260.26	45989.54	na	32.87	37.90	11323.91
DW4	90313.13	5945.33	2952.91	58783.61	na	32.27	37.19	14764.56
DW3	61.82	43.75	BDL	BDL	na	32.93	37.82	BDL
DW2	157.99	41.86	BDL	BDL	na	32.68	37.56	BDL
DW1	135.18	46.53	3.51	71.81	na	32.95	37.78	17.03

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Table G.2

Vial #	Expect Mass (calc) (dpm)	96hr Count (dpm)	96 Obs Counts (dpm/ml)	96 Obs Counts (total dpm)	96 Est Tiss Count (dpm/g)	W/O Water (g)	W/ Water (g)	Mass Removed (dpm)	Expect Mass (calc) (dpm)	120hr Count (dpm)
DW21	118156.37	11695.24	5827.87	120188.09	na	33.07	38.04	29197.61	90990.48	7753.54
DW20	120272.73	11545.72	5753.11	116391.09	na	32.54	37.46	28708.00	87683.09	7460.36
DW19	109545.21	11635.69	5798.09	115132.70	na	32.21	37.20	29280.36	85852.34	7566.61
DW18	13054.79	1094.81	527.65	10407.92	na	31.95	37.05	2664.64	7743.28	688.44
DW17	15300.10	1024.52	492.51	9957.00	na	32.48	37.44	2497.01	7459.99	495.73
DW16	15950.15	1398.79	679.64	13784.49	na	32.51	37.57	3472.97	10311.52	663.11
DW15	8731.06	655.93	308.21	6248.98	na	32.43	37.34	1565.71	4683.27	348.30
DW14	7196.01	517.27	238.88	4837.59	na	32.48	37.43	1218.29	3619.29	235.06
DW13	8431.17	823.41	391.95	7813.55	na	32.16	37.09	1991.11	5822.44	259.72
DW12	381886.24	29907.29	14933.89	299603.73	na	32.26	37.22	75565.49	224038.24	14610.54
DW11	311378.29	22405.79	11183.14	228382.11	na	32.89	37.95	56139.37	172242.74	11370.00
DW10	340920.69	24031.77	11996.13	244673.09	na	32.82	37.82	60700.42	183972.67	12496.00
DW9	188625.83	12114.91	6037.70	120295.16	na	32.23	37.27	30490.39	89804.77	6060.53
DW8	236142.78	18002.26	8981.38	179483.82	na	32.31	37.34	45266.14	134217.69	8703.26
DW7	237484.47	16376.84	8168.67	164704.82	na	32.63	37.62	41088.39	123616.43	9233.94
DW6	42072.86	3282.67	1621.58	32368.38	na	32.36	37.26	8156.55	24211.83	1574.49
DW5	34665.63	2404.55	1182.52	24084.41	na	32.87	37.88	5948.08	18136.33	1360.68
DW4	44019.05	2567.97	1264.23	25065.91	na	32.14	37.08	6384.37	18681.55	1137.91
DW3	BDL	56.48	8.49	172.84	na	32.77	37.61	42.86	129.99	55.56
DW2	BDL	61.49	10.99	220.93	na	32.49	37.43	55.73	165.20	59.07
DW1	54.78	62.56	11.53	235.51	na	32.75	37.75	57.98	177.53	58.95

Table G.2

Vial #	120 Obs Counts (dpm/ml)	120 Obs Counts (total dpm)	120 Est Tiss Count (dpm/g)	W/O Water (g)	W/ Water (g)	Mass Removed (dpm)	Expect Mass (calc) (dpm)	144hr Count (dpm)
DW21	3857.02	79388.97	na	32.99	38.00	19477.93	59911.03	5453.87
DW20	3710.43	74805.91	na	32.39	37.27	18811.86	55994.04	5517.09
DW19	3763.55	74507.03	na	32.14	37.71	19043.57	55463.46	5026.35
DW18	324.47	6416.32	na	32.03	37.03	1628.82	4787.50	435.20
DW17	228.11	4586.63	na	32.39	37.24	1151.96	3434.67	308.89
DW16	311.80	6308.36	na	32.42	37.09	1605.78	4702.59	353.26
DW15	154.40	3104.14	na	32.26	37.36	784.33	2319.80	189.15
DW14	97.78	1965.40	na	32.39	37.31	492.79	1472.61	143.64
DW13	110.11	2178.45	na	32.07	37.10	552.73	1625.72	202.17
DW12	7285.52	145433.48	na	32.20	37.18	36573.29	108860.19	7888.75
DW11	5665.25	115922.27	na	32.86	37.86	28836.10	87086.17	6065.77
DW10	6228.25	126657.62	na	32.79	37.82	31328.08	95329.54	6970.97
DW9	3010.51	59951.32	na	32.23	37.21	15172.98	44778.35	3771.80
DW8	4331.88	86524.90	na	32.29	37.44	21875.98	64648.92	4648.39
DW7	4597.22	92509.78	na	32.52	37.38	23445.80	69063.98	5264.86
DW6	767.49	15220.12	na	32.25	37.34	3845.13	11374.99	947.10
DW5	660.59	13440.95	na	32.81	37.65	3349.17	10091.78	854.60
DW4	549.20	10828.60	na	31.99	36.93	2795.43	8033.17	617.61
DW3	8.03	161.79	na	32.55	37.51	40.61	121.17	57.21
DW2	9.78	195.33	na	32.43	37.44	48.91	146.43	61.30
DW1	9.72	198.33	na	32.71	37.58	49.00	149.34	64.52

Table G.2

Vial #	144 Obs Counts (dpm/ml)	144 Obs Counts (total dpm)	144 Est Tiss Count (dpm/g)	W/O * Water (g)	Mass associated w/ aqueous phase within core (dpm)	W/ * Methanol (g)	Methanol Volume (mL)
DW21	2707.18	55613.63	na	16.24	na	32.07	20.01
DW20	2738.79	54696.40	na	16.12	na	32.07	20.16
DW19	2493.42	50633.91	na	16.03	na	32.00	20.19
DW18	197.85	3908.45	na	15.87	na	31.65	19.95
DW17	134.69	2681.30	na	16.05	na	31.76	19.86
DW16	156.88	3098.62	na	15.94	na	31.72	19.95
DW15	74.82	1505.81	na	15.83	na	31.77	20.15
DW14	52.07	1040.34	na	15.88	na	32.10	20.51
DW13	81.33	1609.95	na	15.83	na	31.67	20.03
DW12	3924.62	78186.31	na	15.88	na	31.71	20.01
DW11	3013.13	61383.51	na	16.09	na	32.19	20.35
DW10	3465.73	70479.11	na	16.05	na	32.22	20.44
DW9	1866.15	37050.47	na	15.91	na	31.78	20.06
DW8	2304.44	46259.36	na	15.95	na	31.63	19.82
DW7	2612.68	51947.84	na	16.06	na	31.99	20.14
DW6	453.80	9035.54	na	15.98	na	31.68	19.85
DW5	407.55	8198.61	na	16.13	na	32.11	20.20
DW4	289.05	5655.87	na	15.98	na	31.70	19.87
DW3	8.85	177.53	na	16.03	na	31.95	20.13
DW2	10.90	217.71	na	16.10	na	32.09	20.21
DW1	12.51	253.03	na	16.14	na	31.88	19.90

Table G.2

Vial #	MeOH Count (dpm)	Methanol Conc. (dpm/mL)	Total MeOH (dpm)	Corrected Extractable ** (dpm)	Combust Count (dpm)	Corrected Combust Count *** (dpm)	Weight Combust (g)	Combust Conc. (dpm/g)
DW21	185.24	72.87	1458.28	na	na	na	na	na
DW20	205.38	82.94	1672.36	na	na	na	na	na
DW19	185.46	72.98	1473.37	na	na	na	na	na
DW18	42.74	1.62	32.24	na	na	na	na	na
DW17	38.58	-0.46	-9.21	na	na	na	na	na
DW16	40.43	0.46	9.20	na	na	na	na	na
DW15	38.12	-0.69	-13.98	na	na	na	na	na
DW14	41.34	0.92	18.79	na	na	na	na	na
DW13	88.28	24.39	488.34	na	na	na	na	na
DW12	96.05	28.27	565.78	na	na	na	na	na
DW11	80.58	20.53	417.96	na	na	na	na	na
DW10	115.46	37.98	776.33	na	na	na	na	na
DW9	55.13	7.81	156.72	na	na	na	na	na
DW8	66.76	13.63	270.11	na	na	na	na	na
DW7	67.98	14.24	286.71	na	na	na	na	na
DW6	47.73	4.11	81.60	na	na	na	na	na
DW5	47.65	4.07	82.25	na	na	na	na	na
DW4	52.51	6.50	129.20	na	na	na	na	na
DW3	40.47	BDL	BDL	na	na	na	na	na
DW2	43.02	BDL	BDL	na	na	na	na	na
DW1	43.15	BDL	BDL	na	na	na	na	na

Table G.2

Vial #	Spiked mg/mL 0.00	24 hr Tissue (mg/g) 24.00	24 hr Water (mg/mL) 24.00	48 hr Tissue (mg/g) 48.00	48 hr Water (mg/mL) 48.00	72 hr Tissue (mg/g) 72.00	72 hr Water (mg/mL) 72.00	96 hr Tissue (mg/g) 96.00	96 hr Water (mg/mL) 96.00	120 hr Tissue (mg/g) 120.00	120 hr Water (mg/mL) 120.00
DW21	2.55E-05	na	2.39E-05	na	1.66E-05	na	1.24E-05	na	9.48E-06	na	6.28E-06
DW20	2.57E-05	na	2.28E-05	na	1.61E-05	na	1.27E-05	na	9.36E-06	na	6.04E-06
DW19	2.56E-05	na	2.32E-05	na	1.63E-05	na	1.21E-05	na	9.43E-06	na	6.12E-06
DW18	1.14E-02	na	1.10E-02	na	5.37E-03	na	3.08E-03	na	1.84E-03	na	1.13E-03
DW17	1.12E-02	na	1.12E-02	na	7.09E-03	na	3.51E-03	na	1.71E-03	na	7.94E-04
DW16	1.11E-02	na	1.11E-02	na	7.37E-03	na	3.62E-03	na	2.36E-03	na	1.08E-03
DW15	5.51E-03	na	5.90E-03	na	3.50E-03	na	1.99E-03	na	1.07E-03	na	5.37E-04
DW14	5.53E-03	na	6.05E-03	na	3.02E-03	na	1.63E-03	na	8.31E-04	na	3.40E-04
DW13	5.63E-03	na	7.28E-03	na	3.95E-03	na	1.97E-03	na	1.36E-03	na	3.83E-04
DW12	9.90E-04	na	9.93E-04	na	5.25E-04	na	2.77E-04	na	1.64E-04	na	7.98E-05
DW11	9.79E-04	na	7.82E-04	na	4.18E-04	na	2.21E-04	na	1.22E-04	na	6.20E-05
DW10	9.79E-04	na	9.74E-04	na	5.45E-04	na	2.41E-04	na	1.31E-04	na	6.82E-05
DW9	4.47E-04	na	4.79E-04	na	2.32E-04	na	1.39E-04	na	6.61E-05	na	3.30E-05
DW8	4.42E-04	na	4.54E-04	na	2.65E-04	na	1.71E-04	na	9.84E-05	na	4.74E-05
DW7	4.42E-04	na	4.47E-04	na	3.02E-04	na	1.72E-04	na	8.95E-05	na	5.04E-05
DW6	9.28E-05	na	1.06E-04	na	5.37E-05	na	3.07E-05	na	1.78E-05	na	8.41E-06
DW5	9.22E-05	na	9.69E-05	na	4.85E-05	na	2.48E-05	na	1.30E-05	na	7.24E-06
DW4	9.29E-05	na	9.24E-05	na	6.17E-05	na	3.23E-05	na	1.38E-05	na	6.02E-06
DW3	na	na	****	na	****	na	****	na	****	na	****
DW2	na	na	****	na	****	na	****	na	****	na	****
DW1	na	na	****	na	****	na	****	na	****	na	****

Table G.2

Vial #	144 hr Tissue (mg/g) 144.00	144 hr Water (mg/mL) 144.00	Meoh Extract (mg/g)	Combust (mg/g)	Extract + Comb Final Conc. (mg/g)	Single Point 144 hr Kwood (mL/g)	% Recovery
DW21	na	4.40E-06	na	na	na	na	76.27%
DW20	na	4.46E-06	na	na	na	na	74.55%
DW19	na	4.06E-06	na	na	na	na	75.25%
DW18	na	6.88E-04	na	na	na	na	52.95%
DW17	na	4.69E-04	na	na	na	na	55.80%
DW16	na	5.46E-04	na	na	na	na	58.62%
DW15	na	2.60E-04	na	na	na	na	59.93%
DW14	na	1.81E-04	na	na	na	na	53.92%
DW13	na	2.83E-04	na	na	na	na	70.62%
DW12	na	4.30E-05	na	na	na	na	53.52%
DW11	na	3.30E-05	na	na	na	na	41.64%
DW10	na	3.80E-05	na	na	na	na	50.46%
DW9	na	2.04E-05	na	na	na	na	55.82%
DW8	na	2.52E-05	na	na	na	na	60.79%
DW7	na	2.86E-05	na	na	na	na	62.58%
DW6	na	4.97E-06	na	na	na	na	60.98%
DW5	na	4.46E-06	na	na	na	na	52.96%
DW4	na	3.17E-06	na	na	na	na	53.46%
DW3	na	****	na	na	na	na	na
DW2	na	****	na	na	na	na	na
DW1	na	****	na	na	na	na	na

Table G.2

Vial #	Vial Wt. (g)	With Tissue (g)	Tissue Wt (g)	W/Water Wt (g)	Water Vol (mL)	W/Cap Wt (g)	Cap Weight (g)	Vial/tissue /cap weight (g)
DC21	15.784	16.787	1.00	36.54	19.76	38.07	1.53	18.32
DC20	15.800	16.734	0.93	36.55	19.81	38.08	1.54	18.27
DC19	15.620	16.567	0.95	36.25	19.68	37.73	1.48	18.05
DC18	15.775	16.766	0.99	36.63	19.86	38.15	1.53	18.29
DC17	15.766	16.842	1.08	36.51	19.66	38.02	1.51	18.36
DC16	15.781	16.849	1.07	36.72	19.87	38.23	1.51	18.36
DC15	15.813	16.802	0.99	36.65	19.85	38.19	1.54	18.34
DC14	15.682	16.762	1.08	36.23	19.47	37.73	1.50	18.26
DC13	15.881	16.890	1.01	36.51	19.62	38.03	1.52	18.41
DC12	15.631	16.633	1.00	36.21	19.58	37.75	1.54	18.17
DC11	15.638	16.640	1.00	36.21	19.57	37.72	1.52	18.16
DC10	15.841	16.837	1.00	36.59	19.75	37.07	0.48	17.32
DC9	15.730	16.701	0.97	36.41	19.70	37.89	1.48	18.18
DC8	15.688	16.701	1.01	36.16	19.46	37.69	1.53	18.23
DC7	15.706	16.653	0.95	36.39	19.74	37.90	1.51	18.16
DC6	15.731	16.665	0.93	36.19	19.52	37.66	1.47	18.14
DC5	15.796	16.828	1.03	36.59	19.76	38.07	1.48	18.31
DC4	15.715	16.700	0.98	36.38	19.68	37.87	1.49	18.19
DC3	15.802	16.719	0.92	36.42	19.70	37.95	1.53	18.25
DC2	15.782	16.779	1.00	36.59	19.81	38.06	1.47	18.25
DC1	15.770	16.751	0.98	36.49	19.74	37.99	1.50	18.25

Table G.2

Vial #	Spiked Counts (dpm/mL)	Spiked Counts (total dpm)	24hr Count (dpm)	24 Obs Counts (dpm/ml)	24 Obs Counts (total dpm)	24 Est Tiss Count (dpm/g)	W/O Water (g)	W/ Water (g)
DC21	16429.72	324569.21	1271.58	616.04	12169.80	311465.02	33.10	37.95
DC20	16383.28	324569.21	1379.77	670.13	13275.97	333290.41	33.12	37.97
DC19	16491.50	324569.21	2055.55	1008.02	19838.87	321784.95	32.63	37.57
DC18	3311.37	65767.11	3666.03	1813.26	36013.18	30024.14	33.14	38.01
DC17	3344.54	65767.11	3705.10	1832.80	36040.11	27627.33	33.06	37.92
DC16	3309.87	65767.11	3674.69	1817.59	36115.54	27763.64	33.21	38.09
DC15	1647.76	32708.06	1947.98	954.24	18941.59	13919.58	33.17	38.05
DC14	1680.09	32708.06	2021.86	991.18	19296.22	12418.37	32.73	37.54
DC13	1666.74	32708.06	2044.83	1002.66	19676.23	12915.59	33.06	37.92
DC12	94156.65	1843210.63	104509.51	52235.00	1022552.39	819020.20	32.75	37.53
DC11	94199.96	1843210.63	110154.99	55057.74	1077314.82	764367.07	32.77	37.64
DC10	93331.85	1843210.63	112713.40	56336.95	1112598.35	733546.46	33.03	37.93
DC9	41834.49	824306.87	48946.13	24453.31	481828.05	352707.34	32.85	37.75
DC8	42356.86	824306.87	49209.05	24584.77	478444.23	341424.12	32.56	37.43
DC7	41764.55	824306.87	51213.07	25586.78	505006.30	337170.61	32.89	37.73
DC6	8791.51	171627.92	10362.46	5161.48	100762.34	75873.21	32.67	37.70
DC5	8686.06	171627.92	10218.05	5089.27	100558.91	68865.32	33.06	37.91
DC4	8719.60	171627.92	8661.28	4310.89	84851.18	88098.22	32.95	37.85
DC3	0.00	0.00	38.24	BDL	BDL	BDL	32.98	37.89
DC2	0.00	0.00	38.80	BDL	BDL	BDL	33.00	37.88
DC1	0.00	0.00	41.27	BDL	BDL	BDL	32.91	37.82

Table G.2

Vial #	Mass Removed (dpm)	Expect Mass (calc) (dpm)	48hr Count (dpm)	48 Obs Counts (dpm/ml)	48 Obs Counts (total dpm)	48 Est Tiss Count (dpm/g)	W/O Water (g)	W/ Water (g)	Mass Removed (dpm)
DC21	3062.32	321506.90	874.37	417.43	8195.85	312373.93	32.95	37.85	2087.16
DC20	3326.53	321242.68	1065.01	512.75	10099.66	333129.57	32.97	37.86	2563.76
DC19	5141.92	319427.30	1344.71	652.60	12738.73	323852.76	32.62	37.57	3230.36
DC18	9091.69	56675.42	2881.52	1421.01	28017.98	28917.69	33.03	37.92	7076.61
DC17	9088.84	56678.27	2719.02	1339.76	26212.33	28314.07	32.91	37.83	6712.18
DC16	9129.76	56637.35	2707.15	1333.82	26312.29	28394.25	33.11	38.00	6642.43
DC15	4793.13	27914.93	1503.41	731.95	14424.56	13640.41	33.04	37.92	3667.08
DC14	4951.92	27756.14	1549.42	754.96	14557.07	12221.37	32.56	37.43	3759.68
DC13	4983.23	27724.83	1532.15	746.32	14563.71	13043.72	32.94	37.85	3716.68
DC12	260966.07	1582244.56	76445.63	38203.06	739611.27	840951.39	32.55	37.50	190251.25
DC11	272700.99	1570509.63	80527.15	40243.82	784110.61	784829.36	32.63	37.56	201621.54
DC10	227657.60	1615553.03	76273.63	38117.06	785516.40	833370.11	32.95	37.84	189822.97
DC9	123195.78	701111.09	36615.61	18288.05	357824.01	353539.73	32.75	37.67	91440.26
DC8	126169.05	698137.82	34729.15	17344.82	333003.22	360448.76	32.41	37.36	87071.00
DC7	128164.19	696142.68	36580.96	18270.73	357521.57	357572.45	32.73	37.70	91353.63
DC6	25740.28	145887.64	6955.92	3458.21	67659.81	83755.71	32.74	37.67	17152.70
DC5	25476.89	146151.03	7034.92	3497.71	68565.54	75179.74	32.91	37.82	17488.53
DC4	21226.80	150401.12	7158.70	3559.60	69978.10	81647.73	32.85	37.72	17797.98
DC3	BDL	BDL	42.74	BDL	BDL	na	32.90	37.75	BDL
DC2	BDL	BDL	47.22	3.86	75.72	na	32.89	37.75	19.24
DC1	BDL	BDL	44.51	2.50	48.95	na	32.88	37.74	12.36

Table G.2

Vial #	Expect Mass (calc) (dpm)	72hr Count (dpm)	72 Obs Counts (dpm/ml)	72 Obs Counts (total dpm)	72 Est Tiss Count (dpm/g)	W/O Water (g)	W/ Water (g)	Mass Removed (dpm)
DC21	319419.74	608.70	284.60	5559.30	312921.67	32.87	37.83	1417.29
DC20	318678.92	468.11	285.74	5596.74	335205.77	32.89	37.97	1420.11
DC19	316196.93	1000.80	480.65	9382.22	323985.97	32.56	37.50	2408.04
DC18	49598.81	2157.70	1059.10	20786.88	29073.58	32.93	37.94	5284.89
DC17	49966.09	1969.97	965.23	18797.88	28966.74	32.85	37.85	4806.85
DC16	49994.92	2053.14	1006.82	19770.85	28299.69	33.04	38.05	4993.81
DC15	24247.85	1017.60	489.05	9574.06	14837.00	32.93	37.89	2440.34
DC14	23996.46	1060.80	510.65	9790.11	13154.03	32.47	37.50	2532.81
DC13	24008.15	1095.93	528.21	10270.54	13615.08	32.83	37.83	2651.62
DC12	1391993.31	54679.77	27320.13	528098.14	862170.83	32.51	37.51	136327.46
DC11	1368888.09	54904.60	27432.55	532301.13	834917.12	37.58	37.57	-548.65
DC10	1425730.06	52361.64	26161.07	536772.76	892527.41	32.87	37.90	130020.50
DC9	609670.83	26400.05	13180.27	256830.77	363378.03	32.72	37.66	65242.34
DC8	611066.82	24919.51	12440.00	237964.79	368313.95	32.40	37.44	61702.41
DC7	604789.05	25656.94	12808.72	250256.70	374374.18	32.71	37.63	63915.49
DC6	128734.93	4860.49	2410.49	47088.95	87415.40	32.66	37.62	12076.56
DC5	128662.50	5041.30	2500.90	48799.99	77386.15	32.83	37.83	12479.47
DC4	132603.13	3723.81	2456.20	47967.16	85924.84	32.69	37.64	12354.69
DC3	BDL	43.03	BDL	BDL	na	32.78	37.70	BDL
DC2	BDL	45.00	2.75	53.57	na	32.75	37.62	13.73
DC1	BDL	48.18	4.34	84.51	na	32.86	37.73	21.16

Table G.2

Vial #	Expect Mass (calc) (dpm)	96hr Count (dpm)	96 Obs Counts (dpm/ml)	96 Obs Counts (total dpm)	96 Est Tiss Count (dpm/g)	W/O Water (g)	W/ Water (g)	Mass Removed (dpm)	Expect Mass (calc) (dpm)	120hr Count (dpm)
DC21	318002.45	481.06	220.78	4308.23	312755.95	32.80	37.77	1110.50	316891.94	350.79
DC20	317258.81	471.42	215.96	4253.69	335123.25	32.94	37.88	1086.26	316172.55	395.94
DC19	313788.89	646.30	303.40	5901.06	325119.15	32.50	37.51	1516.98	312271.91	483.04
DC18	44313.91	1555.00	757.75	14887.44	29693.72	32.91	37.79	3811.46	40502.45	1002.31
DC17	45159.24	1379.29	669.89	13059.53	29832.44	32.80	37.83	3382.95	41776.29	886.47
DC16	45001.11	1103.53	709.35	13964.90	29060.12	33.00	37.94	3582.20	41418.91	1023.33
DC15	21807.51	716.12	338.31	6612.87	15363.64	32.88	37.68	1694.91	20112.60	468.78
DC14	21463.65	785.29	372.89	7175.18	13230.07	32.45	37.52	1883.10	19580.55	507.42
DC13	21356.53	712.89	336.69	6539.89	14684.48	32.78	37.79	1700.29	19656.24	474.28
DC12	1255665.86	35849.59	17905.04	346283.50	907567.22	32.48	37.38	90062.36	1165603.50	24483.57
DC11	1369436.74	41016.73	20488.61	397765.90	969731.38	32.54	37.33	103057.72	1266379.02	28060.72
DC10	1295709.56	34941.86	17451.17	359110.26	940360.75	32.86	37.89	87953.92	1207755.64	23779.46
DC9	544428.49	18340.60	9150.55	178216.04	377149.79	32.61	37.55	46210.26	498218.23	12294.01
DC8	549364.41	16784.27	8372.38	160825.07	383553.15	32.38	37.42	42364.25	507000.16	10777.56
DC7	540873.56	18671.60	9316.05	181364.79	379629.11	32.60	37.47	46859.71	494013.84	12287.98
DC6	116658.37	3251.14	1605.82	31289.33	91401.54	32.57	37.48	8109.37	108549.00	2194.86
DC5	116183.02	3261.13	1610.81	31447.87	82107.71	32.83	37.81	8054.06	108128.97	2114.45
DC4	120248.44	3485.55	1723.02	33511.04	88058.27	32.60	37.45	8684.03	111564.41	2223.72
DC3	BDL	49.55	5.02	97.69	na	32.67	37.69	25.26	BDL	48.90
DC2	BDL	55.42	7.96	154.15	na	32.56	37.43	40.26	BDL	49.35
DC1	BDL	50.09	5.29	103.07	na	32.72	37.53	26.51	BDL	48.31

Table G.2

Vial #	120 Obs Counts (dpm/ml)	120 Obs Counts (total dpm)	120 Est Tiss Count (dpm/g)	W/O Water (g)	W/ Water (g)	Mass Removed (dpm)	Expect Mass (calc) (dpm)	144hr Count (dpm)
DC21	155.64	3027.85	312925.32	32.73	37.66	784.43	316107.51	282.41
DC20	178.22	3494.29	334773.30	32.83	37.80	899.99	315272.56	318.30
DC19	221.77	4315.57	325191.49	32.38	37.31	1137.66	311134.25	565.77
DC18	481.40	9385.88	31399.16	32.75	37.59	2426.26	38076.19	689.82
DC17	423.48	8247.30	31160.77	32.79	37.70	2134.35	39641.94	672.73
DC16	491.91	9630.15	29764.76	32.89	37.81	2484.15	38934.76	782.00
DC15	214.64	4150.42	16139.71	32.66	37.64	1077.47	19035.12	348.81
DC14	233.96	4506.47	13957.49	32.48	37.43	1179.14	18401.41	367.79
DC13	217.39	4213.82	15304.68	32.68	37.64	1110.84	18545.40	363.63
DC12	12222.03	234785.22	928960.36	32.37	37.44	61232.38	1104371.12	16120.91
DC11	14010.61	268639.37	995748.16	32.32	37.26	70193.14	1196185.89	20132.37
DC10	11869.98	244141.67	967483.91	32.86	37.89	59705.98	1148049.66	17635.38
DC9	6127.25	118660.35	390893.80	32.52	37.58	30820.07	467398.15	8640.15
DC8	5369.03	103026.25	398789.65	32.35	37.25	27220.96	479779.20	7805.71
DC7	6124.24	118246.76	396797.35	32.45	37.42	30743.67	463270.18	8862.82
DC6	1077.68	20847.65	93898.66	32.48	37.40	5388.38	103160.62	1490.41
DC5	1037.47	20233.80	85169.73	32.81	37.83	5187.36	102941.61	1495.54
DC4	1092.11	21032.88	91910.19	32.42	37.32	5493.29	106071.12	1603.03
DC3	4.70	91.32	na	32.66	37.66	23.62	BDL	45.86
DC2	4.92	94.42	na	32.44	37.38	24.56	BDL	47.63
DC1	4.40	84.85	na	32.53	37.46	22.01	BDL	51.81

Table G.2

Vial #	144 Obs Counts (dpm/ml)	144 Obs Counts (total dpm)	144 Est Tiss Count (dpm/g)	W/O Water (g)	Mass associated w/ aqueous phase within core (dpm)	W/ Methanol (g)	Methanol Volume (mL)
DC21	121.45	2349.35	312819.70	23.66	834.74	33.78	12.79
DC20	139.40	2721.99	334636.58	23.09	886.00	33.64	13.34
DC19	263.13	5067.91	323195.71	23.40	1797.98	33.26	12.47
DC18	325.16	6274.54	32090.46	23.46	2176.60	33.98	13.30
DC17	316.61	6124.85	31149.72	24.93	2560.75	34.04	11.52
DC16	371.25	7219.63	29695.82	24.35	2784.72	34.32	12.60
DC15	154.65	2984.31	16229.34	23.94	1103.90	33.99	12.71
DC14	164.14	3146.92	14124.53	23.33	1078.08	33.68	13.08
DC13	162.06	3117.09	15290.69	23.84	1126.33	33.98	12.82
DC12	8040.70	154944.31	947531.75	23.93	58673.00	33.63	12.26
DC11	10046.43	191927.02	1002254.35	24.29	76855.20	33.64	11.82
DC10	8797.94	180955.95	970977.62	23.67	60116.30	34.07	13.15
DC9	4300.32	83409.03	395457.39	23.58	29581.91	33.70	12.79
DC8	3883.10	73852.70	400717.17	23.22	25313.94	33.52	13.02
DC7	4411.66	84959.68	399483.10	23.52	30294.84	33.47	12.58
DC6	725.45	13975.82	95486.94	23.24	4769.84	33.66	13.17
DC5	728.02	14213.06	85977.27	23.55	4893.73	34.25	13.53
DC4	781.76	14954.31	92504.37	23.09	4995.45	33.82	13.57
DC3	3.18	61.67	na	22.33	na	33.57	14.21
DC2	4.06	77.71	na	23.13	na	33.98	13.72
DC1	6.15	118.16	na	22.68	na	33.75	13.99

Table G.2

Vial #	MeOH Count (dpm)	Methanol Conc. (dpm/mL)	Total MeOH (dpm)	Actual Extractable ** (dpm)	Combust Count (dpm)	Corrected Combust Count *** (dpm)	Weight Combust (g)	Combust Conc. (dpm/g)
DC21	20378.01	10169.25	130104.71	129269.97	74692.27	139481.36	1.03	135418.80
DC20	22694.85	11327.67	151083.35	150197.35	72363.20	135132.03	0.93	145303.25
DC19	20189.24	10074.87	125585.57	123787.59	81672.81	152516.92	0.96	158871.79
DC18	859.04	409.77	5449.74	3273.14	787.10	1469.84	0.99	1484.69
DC17	867.83	414.16	4769.92	2209.17	859.57	1605.17	1.11	1446.10
DC16	1021.75	491.12	6190.24	3405.52	868.18	1621.25	1.07	1515.19
DC15	415.45	187.97	2388.26	1284.36	599.17	1118.90	1.05	1065.62
DC14	465.21	212.85	2785.10	1707.02	571.65	1067.51	1.06	1007.08
DC13	404.59	182.54	2340.04	1213.71	621.29	1160.21	1.06	1094.53
DC12	17905.26	8932.88	109543.49	50870.49	12128.48	22648.89	1.07	21167.19
DC11	23101.05	11530.77	136299.26	59444.06	13243.17	24730.48	1.04	23779.30
DC10	19095.15	9527.82	125270.98	65154.68	10455.31	19524.39	1.04	18773.45
DC9	9173.24	4566.87	58428.18	28846.27	9964.69	18608.20	1.02	18243.33
DC8	8285.40	4122.95	53686.91	28372.97	4986.44	9311.75	1.06	8784.67
DC7	9329.95	4645.22	58432.30	28137.46	6132.13	11451.22	1.00	11451.22
DC6	1458.41	709.45	9345.74	4575.90	1865.55	3483.75	0.98	3554.85
DC5	1539.98	750.24	10148.58	5254.86	1967.79	3674.68	1.07	3434.28
DC4	1535.87	748.18	10149.16	5153.71	1866.86	3486.20	1.02	3417.84
DC3	48.05	4.27	60.70	na	403.17	na	0.96	na
DC2	47.43	3.96	54.34	na	410.00	na	1.06	na
DC1	50.14	5.32	74.40	na	390.50	na	1.03	na

Table G.2

Vial #	Spiked mg/mL	24 hr Tissue (mg/g)	24 hr Water (mg/mL)	48 hr Tissue (mg/g)	48 hr Water (mg/mL)	72 hr Tissue (mg/g)	72 hr Water (mg/mL)	96 hr Tissue (mg/g)	96 hr Water (mg/mL)	120 hr Tissue (mg/g)	120 hr Water (mg/mL)
		24.00	24.00	48.00	48.00	72.00	72.00	96.00	96.00	120.00	120.00
DC21	2.67E-05	3.41E-03	1.00E-06	5.08E-04	6.79E-07	5.09E-04	4.63E-07	5.09E-04	3.59E-07	5.09E-04	2.53E-07
DC20	2.67E-05	3.65E-03	1.09E-06	5.42E-04	8.34E-07	5.45E-04	4.65E-07	5.45E-04	3.51E-07	5.45E-04	2.90E-07
DC19	2.68E-05	3.52E-03	1.64E-06	5.27E-04	1.06E-06	5.27E-04	7.82E-07	5.29E-04	4.94E-07	5.29E-04	3.61E-07
DC18	1.15E-02	1.04E-01	6.31E-03	1.01E-01	4.94E-03	1.01E-01	3.68E-03	1.03E-01	2.64E-03	1.09E-01	1.67E-03
DC17	1.16E-02	9.61E-02	6.38E-03	9.85E-02	4.66E-03	1.01E-01	3.36E-03	1.04E-01	2.33E-03	1.08E-01	1.47E-03
DC16	1.15E-02	9.66E-02	6.32E-03	9.88E-02	4.64E-03	9.85E-02	3.50E-03	1.01E-01	2.47E-03	1.04E-01	1.71E-03
DC15	5.73E-03	4.84E-02	3.32E-03	4.75E-02	2.55E-03	5.16E-02	1.70E-03	5.35E-02	1.18E-03	5.62E-02	7.47E-04
DC14	5.85E-03	4.32E-02	3.45E-03	4.25E-02	2.63E-03	4.58E-02	1.78E-03	4.60E-02	1.30E-03	4.86E-02	8.14E-04
DC13	5.80E-03	4.49E-02	3.49E-03	4.54E-02	2.60E-03	4.74E-02	1.84E-03	5.11E-02	1.17E-03	5.32E-02	7.56E-04
DC12	1.03E-03	8.97E-03	5.72E-04	9.21E-03	4.18E-04	9.44E-03	2.99E-04	9.94E-03	1.96E-04	1.02E-02	1.34E-04
DC11	1.03E-03	8.37E-03	6.03E-04	8.60E-03	4.41E-04	9.14E-03	3.00E-04	1.06E-02	2.24E-04	1.09E-02	1.53E-04
DC10	1.02E-03	8.03E-03	6.17E-04	9.13E-03	4.17E-04	9.78E-03	2.87E-04	1.03E-02	1.91E-04	1.06E-02	1.30E-04
DC9	4.58E-04	3.86E-03	2.68E-04	3.87E-03	2.00E-04	3.98E-03	1.44E-04	4.13E-03	1.00E-04	4.28E-03	6.71E-05
DC8	4.64E-04	3.74E-03	2.69E-04	3.95E-03	1.90E-04	4.03E-03	1.36E-04	4.20E-03	9.17E-05	4.37E-03	5.88E-05
DC7	4.57E-04	3.69E-03	2.80E-04	3.92E-03	2.00E-04	4.10E-03	1.40E-04	4.16E-03	1.02E-04	4.35E-03	6.71E-05
DC6	9.63E-05	8.31E-04	5.65E-05	9.17E-04	3.79E-05	9.57E-04	2.64E-05	1.00E-03	1.76E-05	1.03E-03	1.18E-05
DC5	9.51E-05	7.54E-04	5.57E-05	8.23E-04	3.83E-05	8.48E-04	2.74E-05	8.99E-04	1.76E-05	9.33E-04	1.14E-05
DC4	9.55E-05	9.65E-04	4.72E-05	8.94E-04	3.90E-05	9.41E-04	2.69E-05	9.64E-04	1.89E-05	1.01E-03	1.20E-05
DC3	na	na	****	na	****	na	****	na	****	na	****
DC2	na	na	****	na	****	na	****	na	****	na	****
DC1	na	na	****	na	****	na	****	na	****	na	****

Table G.2

Vial #	144 hr Tissue (mg/g)	144 hr Water (mg/mL)	Meoh Extract (mg/g)	Combust (mg/g)	Extract + Comb Final Conc. (mg/g)	Single Point 144 hr Kwood (mL/g)	% Recovery
	144.00	144.00					
DC21	5.09E-04	1.98E-07	2.06E-04	2.20E-04	0.000435932	2206.212857	85.89%
DC20	5.44E-04	2.27E-07	2.64E-04	2.36E-04	0.000497014	2191.534242	91.34%
DC19	5.26E-04	4.28E-07	2.13E-04	2.58E-04	0.000474687	1108.831167	90.48%
DC18	1.12E-01	1.13E-03	1.92E-02	5.17E-03	0.016651011	14.71924613	55.17%
DC17	1.08E-01	1.10E-03	1.50E-02	5.03E-03	0.012333048	11.19647052	51.59%
DC16	1.03E-01	1.29E-03	2.01E-02	5.27E-03	0.01637498	12.67814697	55.64%
DC15	5.65E-02	5.38E-04	7.91E-03	3.71E-03	0.00845408	15.71266514	54.98%
DC14	4.91E-02	5.71E-04	9.14E-03	3.50E-03	0.008937734	15.65116414	58.24%
DC13	5.32E-02	5.64E-04	7.68E-03	3.81E-03	0.008185345	14.51759802	56.69%
DC12	1.04E-02	8.81E-05	1.12E-03	2.32E-04	0.000803623	9.125153733	49.16%
DC11	1.10E-02	1.10E-04	1.44E-03	2.60E-04	0.000920091	8.361826927	46.27%
DC10	1.06E-02	9.64E-05	1.32E-03	2.06E-04	0.000931182	9.663532289	48.89%
DC9	4.33E-03	4.71E-05	6.27E-04	2.00E-04	0.000535274	11.36467263	55.43%
DC8	4.39E-03	4.25E-05	5.55E-04	9.62E-05	0.00040745	9.580256262	52.02%
DC7	4.38E-03	4.83E-05	6.40E-04	1.25E-04	0.000457867	9.47587668	55.18%
DC6	1.05E-03	7.95E-06	1.04E-04	3.89E-05	9.45121E-05	11.89491245	49.59%
DC5	9.42E-04	7.97E-06	1.04E-04	3.76E-05	9.47692E-05	11.88524131	50.48%
DC4	1.01E-03	8.56E-06	1.09E-04	3.74E-05	9.60707E-05	11.2201472	48.74%
DC3	na	****	na	na	na	na	na
DC2	na	****	na	na	na	na	na
DC1	na	****	na	na	na	na	na

Table G.2

Vial #	Vial Wt. (g)	With Tissue (g)	Tissue Wt (g)	W/Water Wt (g)	Water Vol (mL)	W/Cap Wt (g)	Cap Weight (g)	Vial/tissue /cap weight (g)
DR21	15.620	16.608	0.99	36.26	19.65	37.76	1.50	18.11
DR20	15.892	16.910	1.02	36.80	19.89	38.30	1.50	18.41
DR19	15.702	16.647	0.94	36.46	19.81	37.94	1.48	18.13
DR18	15.819	16.808	0.99	36.60	19.79	38.11	1.51	18.32
DR17	15.845	16.812	0.97	36.68	19.87	38.23	1.54	18.36
DR16	15.792	16.815	1.02	36.56	19.74	38.10	1.55	18.36
DR15	15.799	16.789	0.99	36.41	19.62	37.91	1.50	18.29
DR14	15.679	16.627	0.95	36.29	19.66	37.80	1.52	18.14
DR13	15.646	16.665	1.02	36.11	19.44	37.57	1.46	18.13
DR12	15.820	16.798	0.98	36.56	19.76	38.06	1.50	18.30
DR11	15.731	16.703	0.97	36.47	19.77	37.96	1.49	18.20
DR10	15.717	16.687	0.97	36.32	19.63	37.82	1.50	18.19
DR9	15.729	16.609	0.88	36.48	19.87	38.01	1.53	18.14
DR8	15.890	16.885	1.00	36.62	19.74	38.16	1.54	18.43
DR7	15.797	16.807	1.01	36.55	19.74	38.08	1.53	18.34
DR6	15.863	16.847	0.98	36.79	19.94	38.28	1.49	18.33
DR5	15.813	16.794	0.98	36.67	19.88	38.15	1.48	18.28
DR4	15.714	16.690	0.98	36.49	19.80	38.00	1.51	18.20
DR3	15.680	16.634	0.95	36.24	19.61	37.81	1.56	18.20
DR2	15.715	16.683	0.97	36.47	19.78	37.93	1.47	18.15
DR1	15.949	16.871	0.92	36.89	20.02	38.38	1.49	18.36

Table G.2

Vial #	Spiked Counts (dpm/mL)	Spiked Counts (total dpm)	24hr Count (dpm)	24 Obs Counts (dpm/ml)	24 Obs Counts (total dpm)	24 Est Tiss Count (dpm/g)	W/O Water (g)	W/ Water (g)
DR21	16516.68	324569.21	433.91	197.20	3875.15	324589.13	32.70	37.54
DR20	16314.93	324569.21	456.76	208.63	4150.41	314753.24	33.33	38.25
DR19	16383.28	324569.21	533.90	247.20	4897.21	338277.26	32.96	37.80
DR18	3323.42	65767.11	4233.68	2097.09	41499.24	24537.78	33.09	37.94
DR17	3309.54	65767.11	4193.92	2077.21	41278.24	25324.58	33.08	37.93
DR16	3331.16	65767.11	4276.56	2118.53	41826.07	23402.78	33.11	38.01
DR15	1667.08	32708.06	2226.11	1093.30	21450.57	11371.20	32.93	37.80
DR14	1663.69	32708.06	2297.82	1129.16	22199.21	11085.28	32.82	37.72
DR13	1682.17	32708.06	2203.75	1082.12	21040.77	11449.75	32.48	37.37
DR12	93289.33	1843210.63	123105.88	61533.19	1215772.70	641552.08	33.00	37.92
DR11	93251.57	1843210.63	123786.48	61873.49	1222991.33	638085.69	32.91	37.81
DR10	93883.29	1843210.63	121951.30	60955.90	1196747.11	666457.23	32.81	37.68
DR9	41485.00	824306.87	54691.58	27326.04	542968.34	319702.87	32.99	38.07
DR8	41762.43	824306.87	55685.64	27823.07	549171.68	276517.77	33.15	38.04
DR7	41749.74	824306.87	55652.93	27806.71	549015.71	272565.51	33.03	37.98
DR6	8605.49	171627.92	11969.96	5965.23	118970.47	53513.66	33.28	38.15
DR5	8634.50	171627.92	11881.97	5921.23	117696.32	54976.15	33.16	38.18
DR4	8666.76	171627.92	11950.10	5955.30	117932.73	55015.56	32.78	37.71
DR3	0.00	0.00	38.33	BDL	BDL	BDL	32.83	37.71
DR2	0.00	0.00	37.53	BDL	BDL	BDL	32.87	37.76
DR1	0.00	0.00	41.20	BDL	BDL	BDL	33.37	38.27

Table G.2

Vial #	Mass Removed (dpm)	Expect Mass (calc) (dpm)	48hr Count (dpm)	48 Obs Counts (dpm/ml)	48 Obs Counts (total dpm)	48 Est Tiss Count (dpm/g)	W/O Water (g)	W/ Water (g)	Mass Removed (dpm)
DR21	997.23	323571.98	371.37	165.93	3224.71	324238.13	32.53	37.43	831.32
DR20	1037.50	323531.71	382.55	171.52	3403.15	314468.13	33.27	38.17	854.18
DR19	1231.53	323337.68	406.02	183.26	3604.47	338342.02	32.84	37.73	908.95
DR18	10516.89	55250.22	3162.75	1561.62	30645.26	24878.63	32.94	37.19	7808.11
DR17	10693.46	55073.65	3304.24	1632.37	31951.94	23910.77	32.94	37.84	8145.51
DR16	10577.80	55189.31	3276.60	1618.55	31804.44	22859.11	33.01	37.80	8092.73
DR15	5445.73	27262.33	1689.03	824.76	16090.27	11284.91	32.82	37.70	4107.31
DR14	5627.72	27080.34	1747.39	853.94	16716.76	10932.06	32.68	37.61	4303.86
DR13	5511.24	27196.82	1574.19	767.34	14764.41	12200.59	32.34	37.25	3859.73
DR12	311111.79	1532098.84	91940.14	45950.32	901637.11	644643.89	32.94	37.82	228832.58
DR11	312646.73	1530563.90	96143.82	48052.16	942446.94	605058.60	32.81	37.72	240260.78
DR10	305571.91	1537638.72	94005.25	46982.87	915696.16	641177.89	32.68	37.56	234914.36
DR9	137122.05	687184.82	43228.65	21594.57	430423.00	291774.80	33.13	38.05	106677.18
DR8	139504.85	684802.01	40615.77	20288.13	397931.41	288312.17	33.07	38.01	100832.01
DR7	140451.70	683855.17	41702.14	20831.32	409189.55	271946.16	32.96	37.91	104573.21
DR6	29814.20	141813.72	8648.58	4304.54	85298.69	57433.97	33.19	38.13	21350.50
DR5	29570.63	142057.29	8668.73	4314.61	85873.71	57271.74	33.23	38.02	21357.33
DR4	31092.60	140535.32	8273.02	4116.76	80326.15	61689.72	32.73	37.64	20501.45
DR3	BDL	BDL	47.16	3.83	74.66	BDL	32.69	37.68	19.21
DR2	BDL	BDL	41.99	BDL	BDL	BDL	32.78	37.70	BDL
DR1	BDL	BDL	42.04	BDL	BDL	BDL	32.26	38.37	BDL

Table G.2

Vial #	Expect Mass (calc) (dpm)	72hr Count (dpm)	72 Obs Counts (dpm/ml)	72 Obs Counts (total dpm)	72 Est Tiss Count (dpm/g)	W/O Water (g)	W/ Water (g)	Mass Removed (dpm)
DR21	322740.66	336.33	148.41	2867.90	323757.86	32.44	37.62	740.57
DR20	322677.54	343.20	151.85	3000.64	314024.46	33.22	38.11	751.64
DR19	322428.73	242.87	135.58	2657.14	338382.64	32.72	37.81	679.23
DR18	47442.11	2266.37	1113.43	21014.90	26721.14	32.89	37.83	4787.75
DR17	46928.14	2378.46	1169.48	22786.08	24965.94	32.79	37.86	5905.86
DR16	47096.58	30.93	-4.29	-83.37	46119.21	33.04	38.06	-20.41
DR15	23155.01	1238.62	599.56	11636.79	11634.57	32.74	37.76	2973.80
DR14	22776.48	1218.07	589.28	11470.90	11925.72	32.61	37.55	2946.39
DR13	23337.09	1172.62	566.56	10833.12	12270.82	32.25	37.25	2832.78
DR12	1303266.26	62140.67	31050.58	606169.45	712777.93	32.83	37.75	154942.40
DR11	1290303.12	63330.57	31645.53	617815.71	691859.47	32.73	37.70	157911.20
DR10	1302724.36	65915.75	32938.12	638011.41	685271.08	32.57	37.51	164361.23
DR9	580507.64	32362.02	16161.26	321802.94	293982.61	33.08	38.10	80321.44
DR8	583970.00	27603.21	13781.85	269903.78	315644.45	33.07	38.04	68082.35
DR7	579281.96	29980.27	14970.38	293015.27	283432.36	32.95	37.98	74253.09
DR6	120463.22	6235.39	3097.94	61326.85	60097.94	33.13	38.17	15489.71
DR5	120699.96	6673.30	3316.90	65485.48	56283.87	33.04	37.94	16518.14
DR4	120033.87	5840.23	2900.36	56388.83	65210.09	32.63	37.64	14530.81
DR3	BDL	42.21	BDL	BDL	na	32.70	37.62	BDL
DR2	BDL	44.84	2.67	52.13	na	32.70	37.66	13.33
DR1	BDL	39.79	BDL	BDL	na	33.38	38.47	BDL

Table G.2

Vial #	Expect Mass (calc) (dpm)	96hr Count (dpm)	96 Obs Counts (dpm/ml)	96 Obs Counts (total dpm)	96 Est Tiss Count (dpm/g)	W/O Water (g)	W/ Water (g)	Mass Removed (dpm)	Expect Mass (calc) (dpm)	120hr Count (dpm)
DR21	322000.09	279.10	119.80	2337.71	323544.92	32.62	37.61	598.98	321401.11	214.50
DR20	321925.90	261.42	110.96	2185.95	314086.39	33.13	38.10	552.56	321373.34	195.37
DR19	321749.50	293.60	127.05	2500.14	337830.00	32.79	37.71	637.77	321111.73	203.95
DR18	42654.36	1531.40	745.95	14556.40	28410.48	32.80	37.81	3752.11	38902.25	994.96
DR17	41022.29	1604.06	782.28	15257.52	26644.02	32.81	37.80	3950.50	37071.79	985.22
DR16	47116.99	2102.21	1031.35	20317.62	26196.84	33.02	38.09	5198.01	41918.98	1474.48
DR15	20181.21	853.39	406.94	7922.74	12382.30	32.72	37.67	2050.98	18130.23	543.25
DR14	19830.09	863.12	411.81	7991.51	12487.95	32.54	37.59	2063.15	17766.94	551.46
DR13	20504.31	804.51	382.50	7313.81	12944.55	32.17	36.98	1943.11	18561.20	473.59
DR12	1148323.86	28815.35	19183.90	373165.13	792595.84	32.70	37.85	96878.67	1051445.19	24162.21
DR11	1132391.92	45362.11	22661.30	441963.36	710317.45	32.68	37.41	113759.73	1018632.18	29512.52
DR10	1138363.14	46135.07	23047.78	445283.14	714515.46	32.48	37.53	115930.34	1022432.80	27025.36
DR9	500186.19	23792.95	11876.72	237083.11	298980.77	33.09	38.00	59502.37	440683.82	16000.71
DR8	515887.66	18856.70	9408.60	184540.21	333012.51	32.99	37.79	47513.41	468374.24	11357.12
DR7	505028.87	21842.20	10901.35	214135.15	288013.59	32.92	38.07	55160.81	449868.06	13782.33
DR6	104973.51	4599.48	2279.99	45225.81	60719.21	33.13	38.06	11491.13	93482.38	3116.99
DR5	104181.82	4883.84	2422.17	47627.06	57650.12	32.87	37.94	12280.38	91901.44	3232.26
DR4	105503.06	3823.31	1891.90	36782.35	70410.57	32.59	37.57	9554.10	95948.96	2680.30
DR3	BDL	51.75	6.12	118.89	na	32.59	37.63	30.79	BDL	47.31
DR2	BDL	49.75	5.12	99.93	na	32.60	33.13	25.91	BDL	47.27
DR1	BDL	47.72	4.11	82.59	na	33.48	38.38	20.49	BDL	41.78

Table G.2

Vial #	120 Obs Counts (dpm/ml)	120 Obs Counts (total dpm)	120 Est Tiss Count (dpm/g)	W/O Water (g)	W/ Water (g)	Mass Removed (dpm)	Expect Mass (calc) (dpm)	144hr Count (dpm)
DR21	87.50	1706.53	323577.51	32.53	37.50	444.48	320956.63	209.56
DR20	77.93	1534.55	314183.49	33.04	37.52	394.33	320979.00	204.80
DR19	82.22	1609.81	338097.26	32.62	38.09	418.51	320693.22	209.76
DR18	477.73	9312.80	29918.56	32.76	37.78	2412.52	36489.73	709.11
DR17	472.86	9194.22	28828.93	32.77	37.77	2378.47	34693.32	694.16
DR16	717.49	14156.01	27138.78	33.04	38.04	3623.31	38295.67	973.12
DR15	251.87	4881.01	13383.05	32.65	37.66	1264.39	16865.84	391.49
DR14	255.98	4977.72	13490.74	32.55	37.48	1290.12	16476.82	401.44
DR13	217.04	4091.45	14199.95	31.90	36.94	1102.57	17458.63	335.57
DR12	12061.35	235823.54	833968.97	32.81	37.80	60789.21	990655.98	16546.80
DR11	14736.51	283132.50	756686.92	32.40	37.24	73829.90	944802.29	19439.11
DR10	13492.93	260953.20	785030.52	32.52	37.43	67599.56	954833.23	18272.61
DR9	7980.60	158510.70	320651.27	32.95	37.86	40302.04	400381.78	11789.91
DR8	5658.81	109577.13	360600.12	32.79	37.82	28294.03	440080.21	7917.72
DR7	6871.41	135593.56	311162.87	33.00	38.06	34838.06	415030.00	10063.03
DR6	1538.74	30353.21	64155.66	33.02	38.03	7755.26	85727.12	2255.20
DR5	1596.38	31389.55	61683.88	32.93	37.85	7997.85	83903.59	2170.33
DR4	1320.40	25578.72	72100.66	32.49	37.42	6707.61	89241.35	1943.21
DR3	3.90	75.81	na	32.59	37.56	19.66	BDL	47.99
DR2	3.88	58.15	na	32.48	37.50	2.52	BDL	49.66
DR1	BDL	BDL	na	33.40	38.16	BDL	BDL	48.57

Table G.2

Vial #	144 Obs Counts (dpm/ml)	144 Obs Counts (total dpm)	144 Est Tiss Count (dpm/g)	W/O Water (g)	Mass associated w/ aqueous phase within core (dpm)	W/ Methanol (g)	Methanol Volume (mL)
DR21	85.03	1649.00	323185.86	22.92	536.69	33.23	13.03
DR20	82.65	1579.45	313752.01	23.11	512.41	33.95	13.70
DR19	85.13	1699.04	337559.98	22.94	535.70	33.82	13.75
DR18	334.80	6516.57	30306.53	21.88	1698.11	33.38	14.54
DR17	327.33	6354.71	29305.70	23.41	2159.70	33.76	13.08
DR16	466.81	9186.78	28454.44	22.83	2807.85	33.48	13.46
DR15	175.99	3408.78	13592.99	23.75	1225.08	33.78	12.68
DR14	180.97	3499.17	13689.50	21.98	968.71	33.49	14.55
DR13	148.03	2784.62	14400.40	23.73	1045.84	33.83	12.77
DR12	8253.65	160962.61	848357.23	23.04	51519.26	34.10	13.98
DR11	9699.80	184713.32	781984.54	22.70	58169.71	33.69	13.89
DR10	9116.55	175402.45	803536.89	23.13	58737.94	33.67	13.32
DR9	5875.20	115870.72	323308.02	21.83	30674.43	33.21	14.39
DR8	3939.11	76395.03	365512.75	23.59	26411.71	34.29	13.53
DR7	5011.76	98846.97	313052.51	23.42	33142.78	34.08	13.48
DR6	1107.85	21820.14	64946.12	23.63	7514.52	34.03	13.15
DR5	1065.41	20853.30	64271.45	23.16	6782.41	33.78	13.43
DR4	951.85	18296.49	72689.40	22.98	5987.14	33.97	13.89
DR3	4.24	82.12	na	22.47	na	33.34	13.74
DR2	5.08	98.24	na	20.36	na	32.68	15.58
DR1	4.53	89.74	na	22.52	na	34.04	14.56

Table G.2

Vial #	MeoH Count (dpm)	Methanol Conc. (dpm/mL)	Total MeOH (dpm)	Actual Extractable ** (dpm)	Combust Count (dpm)	Corrected Combust Count *** (dpm)	Weight Combust (g)	Combust Conc. (dpm/g)
DR21	22651.64	11306.07	147364.78	146828.10	59314.84	114884.45	1.01	113746.98
DR20	23045.00	11502.75	157635.61	157123.21	61693.53	119491.63	1.04	114895.80
DR19	23137.10	11548.80	158850.70	158315.00	65147.11	126180.73	0.99	127455.28
DR18	650.12	305.31	4438.71	2740.60	600.76	1163.59	1.00	1163.59
DR17	679.47	319.98	4186.86	2027.16	684.61	1325.99	0.98	1353.05
DR16	906.66	433.58	5837.66	3029.81	656.29	1271.14	1.02	1246.22
DR15	388.41	174.45	2212.07	986.99	522.75	1012.49	1.02	992.64
DR14	368.82	164.66	2395.95	1427.23	509.79	987.39	0.99	997.36
DR13	337.30	148.90	1901.20	855.36	509.00	985.86	1.05	938.92
DR12	12314.20	6137.35	85814.22	34294.96	4875.17	9442.51	1.00	9442.51
DR11	15141.27	7550.88	104910.48	46740.77	4577.35	8865.68	1.02	8691.84
DR10	14376.07	7168.28	95516.67	36778.73	4600.10	8909.74	1.00	8909.74
DR9	8204.66	4082.58	58735.42	28061.00	2804.55	5432.02	0.88	6172.75
DR8	6390.94	3175.72	42958.49	16546.78	2375.02	4600.08	1.02	4509.88
DR7	8235.84	4098.17	55229.42	22086.64	2579.12	4995.39	1.07	4668.59
DR6	1838.50	899.50	11826.50	4311.98	899.26	1741.74	1.01	1724.49
DR5	1835.45	897.97	12056.20	5273.79	998.45	1933.86	1.05	1841.77
DR4	1509.19	734.84	10209.74	4222.60	999.14	1935.19	0.99	1954.74
DR3	55.30	7.90	108.51	na	392.09	na	0.97	na
DR2	54.63	7.56	117.77	na	388.53	na	0.97	na
DR1	47.03	3.76	54.78	na	412.22	na	0.96	na

Table G.2

Vial #	Spiked mg/mL	24 hr Tissue (mg/g) 24.00	24 hr Water (mg/mL) 24.00	48 hr Tissue (mg/g) 48.00	48 hr Water (mg/mL) 48.00	72 hr Tissue (mg/g) 72.00	72 hr Water (mg/mL) 72.00	96 hr Tissue (mg/g) 96.00	96 hr Water (mg/mL) 96.00	120 hr Tissue (mg/g) 120.00	120 hr Water (mg/mL) 120.00
DR21	2.69E-05	3.56E-03	3.21E-07	5.28E-04	2.70E-07	5.27E-04	2.41E-07	5.26E-04	1.95E-07	5.26E-04	1.42E-07
DR20	2.65E-05	3.45E-03	3.39E-07	5.12E-04	2.79E-07	5.11E-04	2.47E-07	5.11E-04	1.81E-07	5.11E-04	1.27E-07
DR19	2.67E-05	3.71E-03	4.02E-07	5.50E-04	2.98E-07	5.51E-04	2.21E-07	5.50E-04	2.07E-07	5.50E-04	1.34E-07
DR18	1.16E-02	8.54E-02	7.30E-03	8.66E-02	5.43E-03	9.30E-02	3.87E-03	9.88E-02	2.60E-03	1.04E-01	1.66E-03
DR17	1.15E-02	8.81E-02	7.23E-03	8.32E-02	5.68E-03	8.69E-02	4.07E-03	9.27E-02	2.72E-03	1.00E-01	1.65E-03
DR16	1.16E-02	8.14E-02	7.37E-03	7.95E-02	5.63E-03	1.60E-01	-1.49E-05	9.11E-02	3.59E-03	9.44E-02	2.50E-03
DR15	5.80E-03	3.96E-02	3.80E-03	3.93E-02	2.87E-03	4.05E-02	2.09E-03	4.31E-02	1.42E-03	4.66E-02	8.76E-04
DR14	5.79E-03	3.86E-02	3.93E-03	3.80E-02	2.97E-03	4.15E-02	2.05E-03	4.34E-02	1.43E-03	4.69E-02	8.91E-04
DR13	5.85E-03	3.98E-02	3.76E-03	4.24E-02	2.67E-03	4.27E-02	1.97E-03	4.50E-02	1.33E-03	4.94E-02	7.55E-04
DR12	1.02E-03	7.03E-03	6.74E-04	7.06E-03	5.03E-04	7.81E-03	3.40E-04	8.68E-03	2.10E-04	9.13E-03	1.32E-04
DR11	1.02E-03	6.99E-03	6.78E-04	6.63E-03	5.26E-04	7.58E-03	3.47E-04	7.78E-03	2.48E-04	8.29E-03	1.61E-04
DR10	1.03E-03	7.30E-03	6.68E-04	7.02E-03	5.15E-04	7.51E-03	3.61E-04	7.83E-03	2.52E-04	8.60E-03	1.48E-04
DR9	4.54E-04	3.50E-03	2.99E-04	3.20E-03	2.37E-04	3.22E-03	1.77E-04	3.27E-03	1.30E-04	3.51E-03	8.74E-05
DR8	4.57E-04	3.03E-03	3.05E-04	3.16E-03	2.22E-04	3.46E-03	1.51E-04	3.65E-03	1.03E-04	3.95E-03	6.20E-05
DR7	4.57E-04	2.99E-03	3.05E-04	2.98E-03	2.28E-04	3.10E-03	1.64E-04	3.15E-03	1.19E-04	3.41E-03	7.53E-05
DR6	9.43E-05	5.86E-04	6.53E-05	6.29E-04	4.71E-05	6.58E-04	3.39E-05	6.65E-04	2.50E-05	7.03E-04	1.69E-05
DR5	9.46E-05	6.02E-04	6.49E-05	6.27E-04	4.73E-05	6.16E-04	3.63E-05	6.31E-04	2.65E-05	6.76E-04	1.75E-05
DR4	9.49E-05	6.03E-04	6.52E-05	6.76E-04	4.51E-05	7.14E-04	3.18E-05	7.71E-04	2.07E-05	7.90E-04	1.45E-05
DR3	na	na	****	na	****	na	****	na	****	na	****
DR2	na	na	****	na	****	na	****	na	****	na	****
DR1	na	na	****	na	****	na	****	na	****	na	****

Table G.2

Vial #	144 hr Tissue (mg/g) 144.00	144 hr Water (mg/mL) 144.00	Meoh Extract (mg/g)	Combust (mg/g)	Extract + Comb Final Conc. (mg/g) 144.00	Single Point 144 hr Kwood (mL/g)	% Recovery
DR21	5.26E-04	1.38E-07	2.37E-04	1.85E-04	0.00043096	3115.402358	82.12%
DR20	5.10E-04	1.34E-07	2.47E-04	1.87E-04	0.000442076	3287.790268	86.70%
DR19	5.49E-04	1.38E-07	2.61E-04	2.07E-04	0.000489793	3536.551728	89.24%
DR18	1.05E-01	1.16E-03	1.54E-02	4.05E-03	0.01373401	11.79091037	56.69%
DR17	1.02E-01	1.14E-03	1.49E-02	4.71E-03	0.01206396	10.59366177	58.39%
DR16	9.90E-02	1.62E-03	1.99E-02	4.34E-03	0.014626874	9.006382714	56.77%
DR15	4.73E-02	6.12E-04	7.55E-03	3.45E-03	0.007026614	11.47603823	61.10%
DR14	4.76E-02	6.30E-04	8.42E-03	3.47E-03	0.00886144	14.07484723	63.76%
DR13	5.01E-02	5.15E-04	6.30E-03	3.27E-03	0.006286304	12.20615468	57.39%
DR12	9.29E-03	9.04E-05	9.40E-04	1.03E-04	0.000489816	5.418374438	54.06%
DR11	8.56E-03	1.06E-04	1.13E-03	9.52E-05	0.00062658	5.897881331	57.77%
DR10	8.80E-03	9.99E-05	1.05E-03	9.76E-05	0.000515885	5.166593708	56.52%
DR9	3.54E-03	6.43E-05	7.31E-04	6.76E-05	0.000416859	6.478116866	64.66%
DR8	4.00E-03	4.31E-05	4.61E-04	4.94E-05	0.000232777	5.395417714	55.01%
DR7	3.43E-03	5.49E-05	5.65E-04	5.11E-05	0.000293682	5.350193562	60.70%
DR6	7.11E-04	1.21E-05	1.28E-04	1.89E-05	6.73822E-05	5.553254399	61.77%
DR5	7.04E-04	1.17E-05	1.26E-04	2.02E-05	8.04716E-05	6.896160124	62.80%
DR4	7.96E-04	1.04E-05	1.13E-04	2.14E-05	6.91024E-05	6.628357917	58.34%
DR3	na	****	na	na	na	na	na
DR2	na	****	na	na	na	na	na
DR1	na	****	na	na	na	na	na

Table G.2

Vial #	Vial Wt. (g)	With Tissue (g)	Tissue Wt (g)	W/Water Wt (g)	Water Vol (mL)	W/Cap Wt (g)	Spiked Counts (dpm/mL)	Spiked Counts (total dpm)
SW21	15.731	na	na	36.29	20.56	37.92	15789.51	324569.21
SW20	15.772	na	na	36.11	20.34	37.75	15957.19	324569.21
SW19	15.849	na	na	36.36	20.51	37.99	15826.47	324569.21
SW18	15.815	na	na	36.39	20.58	38.04	3195.84	65767.11
SW17	15.720	na	na	36.07	20.35	37.72	3231.80	65767.11
SW16	15.728	na	na	36.18	20.45	37.80	3216.47	65767.11
SW15	15.759	na	na	36.26	20.50	37.90	1595.36	32708.06
SW14	15.793	na	na	36.19	20.40	37.67	1603.41	32708.06
SW43	15.833	na	na	36.18	20.35	37.69	1607.59	32708.06
SW12	15.772	na	na	36.02	20.24	37.50	91054.22	1843210.63
SW11	15.849	na	na	36.32	20.47	37.86	90044.49	1843210.63
SW10	15.926	na	na	36.38	20.46	37.88	90106.11	1843210.63
SW9	15.798	na	na	36.36	20.56	37.83	40088.85	824306.87
SW8	15.784	na	na	36.23	20.44	37.71	40326.15	824306.87
SW7	16.012	na	na	36.69	20.68	38.18	39869.74	824306.87
SW6	15.900	na	na	36.53	20.63	38.05	8320.55	171627.92
SW5	15.751	na	na	36.06	20.31	37.56	8449.17	171627.92
SW4	15.809	na	na	36.46	20.65	37.95	8310.88	171627.92
SW3	15.804	na	na	36.39	20.58	37.85	0.00	0.00
SW2	15.945	na	na	36.56	20.62	38.04	0.00	0.00
SW1	15.715	na	na	36.19	20.47	37.68	0.00	0.00

Table G.2

Vial #	24hr Count (dpm)	24 Obs Counts (dpm/ml)	24 Obs Counts (tot dpm)	W/O Water (g)	W/ MeOH (g)	MeOH Volume (mL)	MeOH Count (dpm)	MeOH Conc. (dpm/mL)
SW21	29623.23	14791.86	304061.50	16.73	31.81	19.06	1830.94	895.72
SW20	28965.33	14462.91	294175.62	16.34	31.92	19.70	1073.32	516.91
SW19	28131.08	14045.79	288050.99	16.58	31.94	19.42	1515.49	737.99
SW18	6189.42	3074.96	63279.53	16.31	32.08	19.94	169.11	64.80
SW17	6087.59	3024.04	61539.24	17.06	31.92	18.79	452.92	206.71
SW16	6374.23	3167.36	64763.04	16.11	31.89	19.95	152.75	56.62
SW15	3207.76	1584.13	32477.76	16.31	31.75	19.52	126.57	43.53
SW14	3076.90	1518.70	30979.89	16.48	31.74	19.29	149.92	55.21
SW13	4098.28	2020.39	41280.80	16.64	34.00	19.44	475.74	248.42
SW12	166975.88	83468.19	1689646.50	16.28	31.73	19.53	3778.76	1869.63
SW11	174903.91	87432.20	1789737.16	16.45	31.91	19.54	5298.80	2629.65
SW10	159464.31	79712.40	1630596.88	16.76	32.46	19.85	6256.18	3108.34
SW9	76711.16	38335.83	788261.26	16.63	31.73	19.09	3150.07	1555.28
SW8	79543.09	39751.79	812566.37	16.21	31.53	19.37	1496.61	728.55
SW7	74233.95	37097.22	766985.05	16.76	32.60	20.03	2681.89	1321.19
SW6	15504.26	7732.38	159495.73	16.24	32.09	20.04	322.18	141.34
SW5	16017.63	7989.06	162281.80	16.02	31.55	19.63	253.82	107.16
SW4	14242.79	7101.64	146656.00	16.38	31.95	19.68	421.28	190.89
SW3	134.58	47.54	978.35	16.27	32.08	19.99	63.04	11.77
SW2	70.81	15.65	322.67	16.47	33.48	21.50	68.06	14.28
SW1	52.26	6.38	130.55	16.13	33.30	21.71	93.13	26.81

Table G.2

Vial #	Total MeOH (dpm)	Combust Count (dpm)	Corrected Combust (dpm)	Combust Weight (g)	Combust Conc. (dpm/g)		Spiked Conc. (mg/mL)	24 hr Aq Conc. (mg/mL)
SW21	17076.36	na	na	na	na		2.57E-05	2.41E-05
SW20	10181.29	na	na	na	na		2.60E-05	2.35E-05
SW19	14330.65	na	na	na	na		2.57E-05	2.29E-05
SW18	1291.93	na	na	na	na		1.11E-02	1.07E-02
SW17	3883.26	na	na	na	na		1.12E-02	1.05E-02
SW16	1129.56	na	na	na	na		1.12E-02	1.10E-02
SW15	849.71	na	na	na	na		5.55E-03	5.51E-03
SW14	1065.04	na	na	na	na		5.58E-03	5.28E-03
SW13	4244.00	na	na	na	na		5.59E-03	7.06E-03
SW12	36517.99	na	na	na	na		9.97E-04	9.14E-04
SW11	51396.12	na	na	na	na		9.86E-04	9.58E-04
SW10	61695.17	na	na	na	na		9.87E-04	8.73E-04
SW9	29689.95	na	na	na	na		4.39E-04	4.20E-04
SW8	14110.50	na	na	na	na		4.42E-04	4.35E-04
SW7	26457.23	na	na	na	na		4.37E-04	4.06E-04
SW6	2832.09	na	na	na	na		9.11E-05	8.47E-05
SW5	2103.84	na	na	na	na		9.25E-05	8.75E-05
SW4	3757.40	na	na	na	na		9.10E-05	7.78E-05
SW3	235.18	na	na	na	na		0.00E+00	****
SW2	307.00	na	na	na	na		0.00E+00	****
SW1	581.99	na	na	na	na		0.00E+00	****

Table G.2

Vial #	24 hr Tissue Conc (calc, mg/g)	MeOH Conc (mg/g)	Combust Conc. (mg/g)	24 hr Single Point Kwood (mL/g)	% Recovery (dpm/dpm)
SW21	na	1.46E-06	na	na	98.94%
SW20	na	8.41E-07	na	na	93.77%
SW19	na	1.20E-06	na	na	93.16%
SW18	na	2.25E-04	na	na	98.18%
SW17	na	7.19E-04	na	na	99.48%
SW16	na	1.97E-04	na	na	100.19%
SW15	na	1.51E-04	na	na	101.89%
SW14	na	1.92E-04	na	na	97.97%
SW13	na	7.50E-04	na	na	139.20%
SW12	na	2.05E-05	na	na	93.65%
SW11	na	2.88E-05	na	na	99.89%
SW10	na	3.40E-05	na	na	91.81%
SW9	na	1.70E-05	na	na	99.23%
SW8	na	7.98E-06	na	na	100.29%
SW7	na	1.45E-05	na	na	96.26%
SW6	na	1.55E-06	na	na	94.58%
SW5	na	1.17E-06	na	na	95.78%
SW4	na	2.09E-06	na	na	87.64%
SW3	****	****	****	****	****
SW2	****	****	****	****	****
SW1	****	****	****	****	****

Table G.2

Vial #	Vial Wt. (g)	With Tissue (g)	Tissue Wt (g)	W/Water Wt (g)	Water Vol (mL)	W/Cap Wt (g)	Spiked Counts (dpm/mL)	Spiked Counts (total dpm)
SC21	15.835	16.836	1.00	36.66	19.82	na	16376.67	324569.21
SC20	15.873	16.901	1.03	36.84	19.93	38.38	16282.19	324569.21
SC19	15.888	16.865	0.98	36.73	19.87	38.22	16337.10	324569.21
SC18	15.855	16.833	0.98	36.69	19.85	38.23	3312.54	65767.11
SC17	15.801	16.764	0.96	36.53	19.76	38.05	3327.96	65767.11
SC16	15.836	16.831	0.99	36.56	19.73	38.06	3333.19	65767.11
SC15	15.707	16.661	0.95	36.35	19.69	37.90	1661.24	32708.06
SC14	15.697	16.671	0.97	36.38	19.71	37.90	1659.30	32708.06
SC13	15.655	16.609	0.95	36.44	19.83	37.04	1649.26	32708.06
SC12	15.740	16.711	0.97	36.51	19.80	37.99	93091.45	1843210.63
SC11	15.655	16.651	1.00	36.22	19.57	37.74	94190.33	1843210.63
SC10	15.803	16.754	0.95	36.60	19.84	38.11	92885.03	1843210.63
SC9	15.735	16.726	0.99	36.41	19.69	37.89	41870.62	824306.87
SC8	15.795	16.772	0.98	36.39	19.61	37.87	42026.45	824306.87
SC7	15.719	16.728	1.01	36.59	19.86	38.11	41507.97	824306.87
SC6	15.759	16.738	0.98	36.67	19.93	38.15	8611.97	171627.92
SC5	15.693	16.694	1.00	36.39	19.70	37.94	8713.85	171627.92
SC4	15.634	16.637	1.00	36.33	19.70	37.87	8713.40	171627.92
SC3	15.781	16.749	0.97	36.21	19.46	37.68	0.00	0.00
SC2	15.767	16.769	1.00	36.47	19.70	37.97	0.00	0.00
SC1	15.844	16.839	0.99	36.02	19.18	37.50	0.00	0.00

Table G.2

Vial #	24hr Count (dpm)	24 Obs Counts (dpm/ml)	24 Obs Counts (tot dpm)	W/O Water (g)	W/ MeOH (g)	MeOH Volume (mL)	MeOH Count (dpm)	MeOH Conc. (dpm/mL)
SC21	1271.58	616.04	12209.22	23.57	33.88	13.03	21497.86	10729.18
SC20	1379.77	670.13	13358.40	25.55	34.74	11.62	7445.76	3703.13
SC19	2055.55	1008.02	20026.36	25.21	34.34	11.54	14789.95	7375.22
SC18	3666.03	1813.26	36000.49	25.83	34.22	10.61	4055.56	2008.03
SC17	3705.10	1832.80	36219.72	24.25	33.79	12.06	3914.13	1937.31
SC16	3674.69	1817.59	35862.89	24.99	34.23	11.68	3963.36	1961.93
SC15	1947.98	954.24	18787.96	23.72	33.56	12.44	1982.49	971.49
SC14	2021.86	991.18	19538.07	24.27	33.75	11.98	1991.20	975.85
SC13	2044.83	1002.66	19884.78	24.07	33.76	12.25	1864.44	912.46
SC12	104509.51	52235.00	1034253.03	24.25	33.81	12.09	105787.20	52873.85
SC11	110154.99	55057.74	1077424.94	24.71	33.80	11.49	109035.09	54497.79
SC10	112713.40	56336.95	1117950.36	24.42	33.91	12.00	108799.09	54379.79
SC9	48946.13	24453.31	481412.34	22.63	33.26	13.44	45565.24	22762.87
SC8	49209.05	24584.77	482205.70	24.18	33.86	12.24	36784.64	18372.57
SC7	51213.07	25586.78	508127.89	23.87	33.66	12.38	48468.70	24214.60
SC6	10362.46	5161.48	102863.06	24.44	34.07	12.17	9864.01	4912.25
SC5	10218.05	5089.27	100238.29	23.63	33.97	13.07	9888.19	4924.34
SC4	8661.28	4310.89	84911.53	24.09	34.05	12.59	9747.18	4853.84
SC3	38.24	BDL	#VALUE!	24.22	33.88	12.21	49.43	4.96
SC2	38.80	BDL	#VALUE!	23.64	33.92	13.00	44.29	2.39
SC1	41.27	BDL	#VALUE!	23.86	33.97	12.78	54.38	7.44

Table G.2

Vial #	Total MeOH (dpm)	Combust Count (dpm)	Corrected Combust (dpm)	Combust Weight (g)	Combust Conc. (dpm/g)		Spiked Conc. (mg/mL)	24 hr Aq Conc. (mg/mL)
SC21	139845.52	59231.01	99065.08	1.00	99065.08		2.66E-05	1.00E-06
SC20	43023.68	47390.96	79262.35	1.05	75487.95		2.65E-05	1.09E-06
SC19	85127.40	79747.53	133379.38	1.00	133379.38		2.66E-05	1.64E-06
SC18	21298.79	773.21	1293.21	1.03	1255.54		1.15E-02	6.31E-03
SC17	23365.30	715.98	1197.49	1.01	1185.63		1.16E-02	6.38E-03
SC16	22918.08	715.15	1196.10	1.04	1150.10		1.16E-02	6.32E-03
SC15	12085.30	582.90	974.91	0.97	1005.06		5.78E-03	3.32E-03
SC14	11695.35	629.59	1053.00	1.01	1042.58		5.77E-03	3.45E-03
SC13	11477.82	646.70	1081.62	0.73	1481.67		5.74E-03	3.49E-03
SC12	639031.57	13214.10	22100.85	0.99	22324.09		1.02E-03	5.72E-04
SC11	626276.77	13029.76	21792.54	1.03	21157.81		1.03E-03	6.03E-04
SC10	652420.00	13894.09	23238.15	0.99	23472.88		1.02E-03	6.17E-04
SC9	305903.00	4672.86	7815.45	1.01	7738.07		4.59E-04	2.68E-04
SC8	224837.47	5799.84	9700.35	0.95	10210.90		4.60E-04	2.69E-04
SC7	299697.72	7198.04	12038.87	1.04	11575.84		4.55E-04	2.80E-04
SC6	59804.02	2881.89	4820.02	0.98	4918.39		9.43E-05	5.65E-05
SC5	64371.29	2012.92	3366.65	1.07	3146.40		9.54E-05	5.57E-05
SC4	61117.84	3100.14	5185.05	1.01	5133.71		9.54E-05	4.72E-05
SC3	60.59	417.53	698.33	1.03	677.99		0.00E+00	****
SC2	31.08	438.39	733.22	1.02	718.84		0.00E+00	****
SC1	95.05	417.06	697.54	1.03	677.22		0.00E+00	****

Table G.2

Vial #	24 hr Tissue Conc (calc, mg/g)	MeOH Conc (mg/g)	Combust Conc. (mg/g)	24 hr Single Point Kwood (mL/g)	% Recovery (dpm/dpm)
SC21	0.00050768	1.75E-05	0.00016117	387.4315768	65.10%
SC20	0.000492528	6.02E-06	0.00012281	177.5104041	31.97%
SC19	0.000507134	1.20E-05	0.000217	221.87104	56.97%
SC18	0.105889642	6.99E-03	0.00436812	12.73958756	88.30%
SC17	0.106746991	6.74E-03	0.0041249	13.91672911	91.69%
SC16	0.104561584	6.83E-03	0.00400127	13.3337769	90.46%
SC15	0.050764115	3.38E-03	0.00349669	14.34650161	96.17%
SC14	0.047042389	3.40E-03	0.00362719	13.20517715	97.42%
SC13	0.046764207	3.17E-03	0.00515483	12.816645322	96.05%
SC12	0.009124825	5.79E-04	0.00024451	13.03489735	91.50%
SC11	0.008421043	5.97E-04	0.00023173	11.81799929	93.14%
SC10	0.008352786	5.96E-04	0.00025709	12.61110669	96.80%
SC9	0.003789701	2.49E-04	8.4752E-05	12.94579507	96.08%
SC8	0.003835111	2.01E-04	0.00011184	9.764548284	86.48%
SC7	0.003432099	2.65E-04	0.00012679	12.07482815	98.87%
SC6	0.000769311	5.38E-05	5.3869E-05	12.78902621	96.46%
SC5	0.000781122	5.39E-05	3.4461E-05	13.29665172	97.08%
SC4	0.000946931	5.32E-05	5.6228E-05	15.33433426	86.89%
SC3	****	****	****	****	****
SC2	****	****	****	****	****
SC1	****	****	****	****	****

Table G.2

Vial #	Vial Wt. (g)	With Tissue (g)	Tissue Wt (g)	W/Water Wt (g)	Water Vol (mL)	W/Cap Wt (g)	Spiked Counts (dpm/mL)	Spiked Counts (total dpm)
SR21	15.727	16.719	0.99	36.40	19.68	37.87	16494.01	324569.21
SR20	15.759	16.762	1.00	36.45	19.69	37.94	16488.15	324569.21
SR19	15.736	16.718	0.98	36.40	19.69	37.91	16487.31	324569.21
SR18	15.741	16.722	0.98	36.63	19.90	38.15	3304.38	65767.11
SR17	15.769	16.779	1.01	36.51	19.73	38.01	3332.68	65767.11
SR16	15.730	16.715	0.98	36.40	19.69	37.95	3340.81	65767.11
SR15	15.644	16.654	1.01	36.32	19.67	37.83	1662.84	32708.06
SR14	15.709	16.692	0.98	36.41	19.71	37.86	1659.21	32708.06
SR13	15.672	16.683	1.01	36.48	19.79	37.94	1652.54	32708.06
SR12	15.643	16.628	0.98	36.26	19.63	37.76	93907.21	1843210.63
SR11	15.798	16.785	0.99	36.69	19.91	38.19	92591.08	1843210.63
SR10	15.836	16.825	0.99	36.67	19.84	38.15	92889.72	1843210.63
SR9	15.774	16.789	1.02	36.54	19.75	38.05	41732.83	824306.87
SR8	15.762	16.752	0.99	36.56	19.81	38.09	41610.64	824306.87
SR7	15.803	16.756	0.95	36.50	19.75	38.00	41741.28	824306.87
SR6	15.834	16.857	1.02	36.69	19.83	38.21	8653.65	171627.92
SR5	15.717	16.741	1.02	36.56	19.82	38.12	8658.02	171627.92
SR4	15.838	16.758	0.92	36.64	19.88	38.12	8633.20	171627.92
SR3	15.715	16.731	1.02	35.94	19.21	37.46	0.00	0.00
SR2	15.845	16.830	0.98	36.22	19.39	37.75	0.00	0.00
SR1	15.675	16.691	1.02	36.31	19.62	37.65	0.00	0.00

Table G.2

Vial #	24hr Count (dpm)	24 Obs Counts (dpm/ml)	24 Obs Counts (tot dpm)	W/O Water (g)	W/ MeOH (g)	MeOH Volume (mL)	MeOH Count (dpm)	MeOH Conc. (dpm/mL)
SR21	433.91	197.20	3880.48	22.99	33.61	13.43	23331.14	11645.82
SR20	456.76	208.63	4106.81	23.96	33.99	12.68	20526.51	10243.50
SR19	533.90	247.20	4866.31	23.37	33.60	12.93	22239.73	11100.11
SR18	4233.68	2097.09	41738.31	23.94	33.92	12.62	3297.09	1628.79
SR17	4193.92	2077.21	40991.59	24.01	33.77	12.34	3582.04	1771.27
SR16	4276.56	2118.53	41705.31	23.67	33.76	12.76	3318.76	1639.63
SR15	2226.11	1093.30	21505.24	22.82	33.32	13.27	1629.73	795.11
SR14	2297.82	1129.16	22259.06	23.45	33.44	12.63	1698.52	829.51
SR13	2203.75	4082.12	21448.43	22.72	33.82	14.03	1630.85	795.67
SR12	123105.88	61533.19	1207773.38	22.74	33.47	13.57	88045.59	44003.04
SR11	123786.48	61873.49	1231715.49	23.40	33.92	13.30	89734.49	44847.49
SR10	121951.30	60955.90	1209547.85	22.89	33.72	13.69	86163.23	43061.86
SR9	54691.58	27326.04	539743.87	23.85	33.47	12.16	44404.00	22182.24
SR8	55685.64	27823.07	551174.94	22.57	33.70	14.07	38481.08	19220.79
SR7	55652.93	27806.71	549126.94	21.93	33.51	14.64	36393.30	18176.90
SR6	11969.96	5965.23	118308.33	23.49	33.83	13.07	8534.23	4247.36
SR5	11881.97	5921.23	117376.57	22.36	33.08	13.55	6885.57	3423.03
SR4	11950.10	5955.30	118391.29	22.94	33.92	13.88	8400.69	4180.59
SR3	38.33	BDL	#VALUE!	23.11	33.56	13.21	57.09	8.79
SR2	37.53	BDL	#VALUE!	21.33	33.40	15.26	59.09	9.79
SR1	41.20	BDL	#VALUE!	22.37	33.41	13.96	55.84	8.17

Table G.2

Vial #	Total MeOH (dpm)	Combust Count (dpm)	Corrected Combust (dpm)	Combust Weight (g)	Combust Conc. (dpm/g)		Spiked Conc. (mg/mL)	24 hr Aq Conc. (mg/mL)
SR21	156357.23	47743.68	76931.49	0.96	80136.96		2.68E-05	3.21E-07
SR20	129889.15	57646.71	92888.67	0.98	94784.36		2.68E-05	3.39E-07
SR19	143557.70	61833.72	99635.39	0.95	104879.35		2.68E-05	4.02E-07
SR18	20550.36	737.90	1189.01	0.96	1238.55		1.15E-02	7.30E-03
SR17	21855.32	630.16	1015.40	1.06	957.93		1.16E-02	7.23E-03
SR16	20915.08	692.24	1115.44	1.03	1082.95		1.16E-02	7.37E-03
SR15	10554.58	587.94	947.37	1.00	947.37		5.79E-03	3.80E-03
SR14	10476.32	506.61	816.32	0.90	907.03		5.77E-03	3.93E-03
SR13	11466.66	507.18	847.24	0.99	825.60		5.76E-03	3.76E-03
SR12	596905.98	6416.57	10339.30	1.00	10339.30		1.03E-03	6.74E-04
SR11	596454.63	6290.37	10135.95	0.98	10342.81		1.01E-03	6.78E-04
SR10	589582.75	6209.74	10006.03	0.97	10315.49		1.02E-03	6.68E-04
SR9	269776.47	3758.10	6055.59	1.03	5879.21		4.57E-04	2.99E-04
SR8	270451.77	2980.18	4802.09	0.96	5002.18		4.56E-04	3.05E-04
SR7	266104.25	2821.81	4546.91	0.97	4687.53		4.57E-04	3.05E-04
SR6	55521.77	1093.37	1761.80	1.02	1727.25		9.48E-05	6.53E-05
SR5	46390.51	893.03	1438.98	1.05	1370.46		9.48E-05	6.49E-05
SR4	58031.47	1112.84	1793.17	0.92	1949.10		9.46E-05	6.52E-05
SR3	116.14	419.28	675.60	1.02	662.36		0.00E+00	****
SR2	149.41	402.32	648.28	1.01	641.86		0.00E+00	****
SR1	113.98	418.28	673.99	1.07	629.90		0.00E+00	****

Table G.2

Vial #	24 hr Tissue Conc (calc, mg/g)	MeOH Conc (mg/g)	Combust Conc. (mg/g)	24 hr Single Point Kwood (mL/g)	% Recovery (dpm/dpm)
SR21	0.000525946	1.89E-05	0.00013038	1192.553084	64.08%
SR20	0.000519811	1.67E-05	0.00015421	1064.637839	59.05%
SR19	0.000529668	1.81E-05	0.00017063	1001.838473	64.78%
SR18	0.085216934	5.67E-03	0.004430901	10.5672436	95.83%
SR17	0.085342277	6.16E-03	0.0033327	10.90913163	96.52%
SR16	0.084987433	5.70E-03	0.00376765	10.55734274	96.27%
SR15	0.0385889477	2.77E-03	0.00329598	10.41622076	99.82%
SR14	0.036981461	2.89E-03	0.0031556	10.17391164	101.63%
SR43	0.038850038	2.77E-03	0.00287496	10.05294644	101.17%
SR12	0.007065688	4.82E-04	0.00011324	10.01886481	98.26%
SR11	0.006785687	4.91E-04	0.00011328	9.932850928	99.53%
SR10	0.007017459	4.72E-04	0.00011298	9.94584036	97.95%
SR9	0.00307065	2.43E-04	6.4393E-05	9.944937479	98.66%
SR8	0.003021727	2.11E-04	5.4787E-05	9.992939161	100.04%
SR7	0.003162582	1.99E-04	5.1341E-05	10.213332973	99.24%
SR6	0.000570859	4.65E-05	1.8918E-05	9.387013471	101.92%
SR5	0.000580268	3.75E-05	1.501E-05	7.888306538	95.94%
SR4	0.000633783	4.58E-05	2.1348E-05	10.91915109	103.44%
SR3	****	****	****	****	****
SR2	****	****	****	****	****
SR1	****	****	****	****	****

Table G.2

Vial #
SR21
SR20
SR19
SR18
SR17
SR16
SR15
SR14
SR13
SR12
SR11
SR10
SR9
SR8
SR7
SR6
SR5
SR4
SR3
SR2
SR1

Table G.2

Notes:

Vial # Notes: D - desorption, S - sorption, W - water only, R - Russian Olive + water, C - Cottonwood + water, vials # 1-3 = water + tce only, vials #4-18 = water + tce, vials #19-21 = water + tissue + pyrene

SR5 - added meoh w/o recording empty weight, poured out methanol, weighed empty, then refilled with meoh, meoh count may be low because of meoh initially poured out.

DC14 - w/o water weight (taken before adding meoh) may be in error (or caused an error), the w/o water weight is a "new" weight after adding meoh & pouring off due to a spill.

DW19-21 - are high possibly due to contamination between steps, highest TCE concentrations were completed immediately prior to the pyrene samples, without rinsing between. Rinsing resumed on the 96 hr sampling day.

SW13 - shows an increase in TCE mass over 24hr period on a water control sample, assumed contamination - will not include in overall calculations

* measured without the aluminum cap

** Extractable minus aqueous phase TCE in tissue

*** Count (incorporates known recovery efficiency measured using blanks)

**** Control samples which did not contain any spiked TCE; specific activity of any counts detected above background is unknown because source of potential contamination is also unknown; use raw data from previous cells as a qualitative measure of the cleanliness of the procedure.

Table G.3 Lipid extractions from trees at Hill Air Force Base site OU-4

Sample	thimble tare wt (g)-	thimble + core wt (g)	thimble + oil + (core) wt (g)	thimble + (oil)+ Na₂SO₄ + core wt (g)	weight of core (g)	weight of oil (g)	weight of Na₂SO₄	rbf (g) *	rbf-end of day (g)	rbf-dried over night (g)	lipid weight (g)	lipid fraction	% spike recovery
C1 (box elder)	1.962	4.475	4.475	9.512	2.513	0	5.037	111.485	111.498	111.495	0.01	0.00398	na
C2 (apple)	2.07	5.355	5.355	11.686	3.285	0	6.331	111.545	111.559	111.561	0.016	0.00487	na
C3 (cottonwood)	2.049	4.683	4.683	10.456	2.634	0	5.773	110.568	110.583	110.587	0.019	0.00721	na
C4 (russian olive)	2.054	4.717	4.717	10.869	2.663	0	6.152	100.866	100.882	100.877	0.011	0.00413	na
C10 (poplar)	2.017	4.69	4.69	9.718	2.673	0	5.028	113.564	113.592	113.587	0.023	0.00860	na
6 (matrix spike) c10 + veg oil spike	2.006	4.763	4.861	11.425	2.757	0.098	6.564	112.911	113.023	112.019	-0.892	**	**
7 (blank spike)	2.109	2.109	2.216	7.023	0	0.107	4.807	108.991	109.095	109.095	0.104	na	97.20%
8 (blank)	2.024	2.024	2.024	7.217	0	0	5.193	111.055	111.063	111.055	0	na	na

* rbf - round bottom flask   ** Invalid results due to laboratory error